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MINUTES  
of the Sixth  
EXPLOSIVES SAFETY SEMINAR  
on  
HIGH-ENERGY SOLID PROPELLANTS

Held at the  
Barksdale Air Force Base  
Shreveport, La.

on  
18-20 August 1964.

~~Host~~  
Longhorn Army Ammunition Plant  
Marshall, Texas

Armed Services Explosives Safety Board,  
Washington, D. C. ~~20333~~

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FOR THE CHAIRMAN:

EDWARD H. HILSMAN  
Colonel, USA

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## PREFACE

This is a record of the proceedings of the Sixth Annual Explosives Safety Seminar on High Energy Solid Propellants held at Barksdale Air Force Base, Shreveport, Louisiana, 18 thru 20 August 1964.

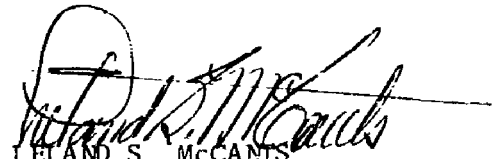
The Armed Services Explosives Safety Board (ASESB) sponsors the annual Seminar as a means of providing an exchange of current information on explosives safety between those segments of Government and industry concerned with high energy propellants. Selected papers are presented by the participants during the course of the Seminar, and a free discussion of the subject matter is encouraged.

The material contained herein represents reports and opinions of the participants, and is a product of the individual or organization which he represents. The ASESB does not vouch for the accuracy of the facts presented, and does not necessarily endorse the opinions expressed.

Rapid and widespread exchange of information concerning explosives incidents and accidents is a vital component of a cooperative effort on the part of Government and industry to develop effective means of prevention in their safety programs. Questions and comments concerning the material herein should be directed to the individual speakers or their organization.

The Armed Services Explosives Safety Board, Nassif Building, Washington, D. C., 20315, should be advised of errors, omissions, or other corrections that may be required in the text.

Appreciation is expressed to all participants for their interest, and their active role in promoting the cause of explosives safety within the Department of Defense and in the industries represented at the Seminar.

  
LELAND S. MCCANTS  
Colonel, USAF  
Chairman, ASESB  
2 October 1964

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Minutes of the  
Sixth Annual  
EXPLOSIVES SAFETY SEMINAR ON HIGH ENERGY SOLID PROPELLANTS

Barksdale Air Force Base  
Shreveport, Louisiana  
18-20 August 1964

Sponsor  
Armed Services Explosives Safety Board

Host  
Longhorn Army Ammunition Plant  
Marshall, Texas



Colonel Leland S. McCants, USAF  
Chairman  
Armed Services Explosives Safety Board

Ladies and gentlemen. The Sixth Annual Explosives Safety Seminar on High Energy Solid Propellants, sponsored by the Armed Services Explosives Safety Board, Department of Defense, Washington, D. C. is called to order.

I am delighted to see so many familiar faces - individuals who join regularly with us. To both old and new friends and participants, allow me to say - many have worked long and hard to make this meeting both challenging and rewarding. We appreciate your participation, one and all.

Our host this year is the United States Army and the Longhorn Army Ammunition Plant operated by the Thiokol Chemical Corp. for the Army. This association has afforded us the distinct privilege of having with us this morning a distinguished officer of the United States Army, Brigadier General Durward E. Breakefield. General Breakefield's military career began with his enlistment in the U. S. Army Air Corps at Brooks Field, Texas on 7 June 1929. He subsequently entered West Point in 1931, and was commissioned in the Field Artillery upon graduation in 1935.

In addition to graduation from the Point, his educational achievements include both the Basic and Advanced Ordnance Courses, the Command and General Staff College, the Army War College, and a Master in Business Administration from the University of Michigan. He served as an instructor and Associate Professor of Ordnance at West Point from 1950 until 1953.

General Breakefield's assignments cover a broad range. He has served as a Battery Officer in the Canal Zone; as Works Manager at Watertown Arsenal; Commanding Officer of the Manila Base Ordnance Center in the Philippines; Chief of Military Personnel in the Office, Chief of Ordnance; and as Chief of the Logistical Division with MAAG, Viet Nam.

His more recent service includes assignment to Letterkenny Ordnance Depot in October 1959 as Commanding Officer, where he was promoted to Brigadier General in June of 1960. From February 1962 until May of this year he was Commanding General of the 8th U. S. Army Support Command, Korea. Since May he has been

assigned as Director of Maintenance, Headquarters, U. S. Army Supply & Maintenance Command at Washington, D. C.

General Breakefield has been awarded the Legion of Merit and the Army Commendation Metal.

General Breakefield.

Brigadier General Durward E. Breakefield, USA  
Director of Maintenance  
US Army Supply & Maintenance Command

Colonel McCants, Members of the Armed Services Explosives Safety Board, and distinguished guests:

It is my pleasure to extend to you the Army's welcome to this Sixth Safety Seminar on High Energy Solid Propellants.

At last year's seminar, Colonel McCants spoke of the birth of the Armed Services Explosives Safety Board in 1928 and traced its growth to its present state of maturity. The Army is proud to have provided the Board's first Chairman, Colonel Miles, who recently passed away and its interest in the work of the Board has never waned. Activities of the Board have been, and will continue to be, actively supported by the Army. One such activity which has had our most enthusiastic support is this annual explosives safety seminar on high energy solid propellants. We have profited greatly from the first five seminars and expect this one to be even more profitable. We welcome this opportunity for freely sharing our knowledge and experiences.

Few people can be expertly informed on the wide variety of subjects discussed at these seminars. However, through meetings such as these, we are able to profit from the knowledge and experience of the experts. Past seminars have provided insight into such subjects as:

- a. Accident investigation techniques and procedures.
- b. Sensitivity and strength of reactions in propellants.
- c. Shock induced detonations in solid explosives charges.
- d. Safety aspects of propellant mixing operations.
- e. Preparation of hazardous propellants by the slurry process.
- f. Dissemination of accident information.
- g. Hazard classification of large solid propellant motors.
- h. Remote operations in research and experimental production of sensitive propellants.

Some of these subjects undoubtedly will come up again for discussion in light of additional knowledge and experience that has been acquired. I am sure that minutes of previous seminars are well worn because of the many references to these and other presentations that are included.

It is important to the success of these seminars, that participants be provided the opportunity to see how others in the solid propellant industry do things. All of us can learn from others. It is for this reason that we are here for this seminar. While you are here, you will have the opportunity to tour one of the most important Army solid propellant facilities - Longhorn Ammunition Plant. Many of you will want to equate what you see and hear during ensuing presentations, to both your home facilities and those at the Longhorn Plant. I hope each of you will take home something that will contribute to increased safety in your operations, because, to paraphrase a statement by President Johnson, "We can afford to spend whatever is necessary for our defense, but we cannot afford any waste due to preventable accidents."

Because I am convinced that what you take away from this seminar will be proportional to what you contribute to it, please be an inquiring and even skeptical audience. Participate energetically and fully in discussions of subjects presented by the various speakers and also in the discussion of your problems and those of your colleagues. Participate actively in the established program of exchanging accident information.

It is an honor for me to be with you today. You, as a group, represent the epitome in safety management. Your record speaks for itself.

And I must say that in my military visits and inspections I have never felt safer than when I was in one of our ammunition plants or storage areas. You know the full meaning of the statement "we cannot afford to have an accident" and the history of explosives manufacture echos this theme.

I congratulate you on your wonderful accomplishments.

Col. McCants: General Breakefield we are most grateful for your participation here today and thank you for your reflected interest in the activities of this professional group. We thank you very much.

Successful mission accomplishment is almost always the result of a dedicated 'team effort' derived from a skillful blending of many talents. We have with us this morning two of the men who are members of such a team. I would like to introduce them: Lt. Col. Russel Whitmore, USA, Commander, Longhorn Army Ammunition Plant whom we would like to thank for all the many services rendered in making this Seminar possible. Col. Whitmore we are deeply appreciative of your efforts. Another member of the team is Mr. R. A. McElvogue, General Manager, Longhorn Division, Thiokol Chemical Corp. Mr. McElvogue we are delighted to have you with us as a partner in this professional effort.

We also have with us the Technical Director, Longhorn Division, Thiokol Chemical Corp. in the person of Mr. John Sims. Mr. Sims has been the Project Officer for this Seminar and has done a splendid job. Mr. Sims has been most ably assisted by one of those 'bright-eyed, bushy-tailed individuals' its a genuine pleasure to be associated with when there are things that must be done and done right. I'm referring of course to none other than Mr. O. C. Wetzell, Security Officer, Longhorn Division, Thiokol Chemical Corp. The team of Sims and Wetzell have produced commendably and we are deeply indebted to them for their splendid efforts. Thank you.

Now I'd like to have the 'Father of the ASES Seminar' make the introductory remarks; a respected professional associate, a valued friend, and a grand gentleman who needs no introduction to this group. Mr. Henry Marsh.

Mr. Henry Marsh  
Senior Vice President  
American Ordnance Association

A hearty welcome to all of you here present for this, the 6th Annual Safety Seminar on High Energy Solid Propellants, in this excellent facility at Barksdale Air Force Base and with the Army as host agency. Our sincere thanks go to both Army and Air Force for what we hope will prove to be an excellent session.

That we have possibilities for help to each of you is proven by your large attendance which seems to increase each year. I see many faces of those who have attended all seminars and some new ones whom I hope to meet.

May I exercise the privilege that goes to those of us with gray hair and increased age and reminisce a wee bit? I'll try not to take you too far back and I'll try to keep my facts straight.

Let's go back to early 1940 and the start of World War II. There were only a few companies who manufactured explosives and propellant for military use and each of them guarded their know-how and process methods jealously and they barely spoke to one another and would release no information to one another. The pressure for tremendous tonnages of these products for U. S. use and for supply to our Allies broke down this attitude and the integration (that's a bad word these days) helped and soon easy exchange of process information developed. This was particularly true in matters that dealt with safety. Some serious accidents with much loss of life that upset some long established beliefs hastened the process. Towards the end of World War II enemy activity forced attention on the guided missile field and we see the beginnings and active pursuit of larger and larger efforts in this field with spectacular break-through to the present rapidly developing effort where intercontinental flights are routine and interplanetary travel is visible in the not too distant future.

Some six years ago, the entry of many new companies and federal agencies in this field made obvious the need for exchange of safety information to protect the lives of those working in the field and to protect the enormous investment of Government funds in the activity and the ASES was urged to set up the first of these seminars. Captain Jenkins, USN, then Chairman, moved promptly and the first seminar was held at Indian Head at the Naval Propellant Plant, then commanded by Capt. Gris Atkins and we had about 50 people present and were most proud of the results.

Subsequently under the leadership of Col. Hamilton, USA, and now under our present energetic Chairman, Col. McCants, USAF, we have held seminars at Redstone with Army as host, then Air Force at Riverside, California and then were guests of NASA at Langley Research Center who joined in our efforts, and last year we started around the circle again with Navy as host at Santa Monica, Calif. and now here at Shreveport with Army as host at an Air Force installation. It is heart-warming to see this excellent cooperation between the three Services now joined by NASA, which shows the intense interest and national importance of the subject.

One point I want to make here. The runaway rate of advancement in this field and in other scientific fields have lead to introduction of many highly technical papers to our programs. That's fine, and we all profit from it, but please, please don't let us forget our primary interest is safety and let's use this seminar to tell others working in the field of our unfortunate experiences in enough detail so that we may help them to avoid similar experience. Don't hold back information that may save a life.

One other reminiscence and I'll quit. I was introduced to you as Senior Vice-President of the American Ordnance Association. As I look around the room, I can see many whom I recognize as members; and all I can say is that there is not a single person here who should not be a member. For the benefit of those who do not belong, let me tell you a little bit about AOA and its objectives. It is a non-profit, non-political organization primarily interested in industrial preparedness as the best way to keep the peace. It has both industrial and military members who, if God forbid, war should come, will be involved in efforts to insure that our fighting men in Army, Navy and Air Force shall have the most modern, up-to-date ordnance to permit them to fight with best chance of victory, and least loss of life.

Let me talk just for a moment about AOA Technical Divisions and Sections which cover the entire field of ordnance. There are 16 of them: Artillery, Bomb, Warhead and Artillery Ammunition, Combat and Tactical Vehicles, Fire Control, Missiles and Astronautics, Small Arms and Small Arms Ammunition, Underwater Ordnance, Critical Components, Electronics, Logistics, Materials, Packaging, Prod. Equipment and Techniques, Research Standards, Meteorology and Technical Documentation. Especially qualified members serve in these divisions and their 85 sections to assist the Services, Bureau of Standards and NASA on armament research, development and production techniques. Problems, many classified, are submitted by the Services and the findings are made available to them.

They have often proved of great value. About 10% of our membership of 45,000 serve in the Technical Divisions. Every man in the room should have an interest in this and almost everyone would be qualified and would be welcome.

I've brought along some literature and membership applications placed on the table. I would welcome a chance to talk with any of you and I'd welcome a handful of applications. The dues are nominal, \$5.00 a year or \$12.50 for three, and a special military rate of \$4.00 a year or \$10.00 for three to Government employees of Services.



Col. McCants: Thank you Mr. Marsh. We have two gentlemen with us who were former Board Chairmen, I would like to introduce them. Col. Andy Hamilton, USA (Ret) and Capt. Walter Jenkins, USN (Ret).

I'd like to express our thanks to the Commander and Staff, Barksdale AFB for the fine facilities and services made available to us. I'd also like to take a moment to recognize the efforts of my staff in arranging the many complexities associated with this Seminar. They've done a fine job and I appreciate it.

Each year we are privileged to have with us a legal advisor from the Office of the General Counsel, OSA. Mr. Bruce Docherty, Assistant General Counsel of that office is our advisor this year. Mr. Docherty will now make such remarks as he considers pertinent to this gathering.

Mr. Bruce M. Docherty  
Assistant General Counsel, OSA

Thank you, Col. McCants. Each year the Armed Services Explosives Safety Board has asked the General Counsel of the Army to make an attorney available for attendance at its Safety Seminar on High Energy Solid Propellants. This is my second appearance in that capacity. Those who have attended prior Seminars will recall that the reasons for attendance of a Government attorney have been explained each year. At the risk of boring a considerable portion of my audience, I will restate those reasons briefly. The President has recognized that information and advice obtained through activities such as this Seminar are beneficial to the operations of the Government. He has prescribed uniform standards for the departments and agencies of the Government to follow in order that committees and similar groups sponsored by the Government shall function at all times in consonance with the antitrust and conflict of interest laws. This Seminar is, of course, being conducted in accordance with the standards applicable to this type of meeting. It is felt, however, that since any such meeting as this is subject to the provisions of the antitrust laws, a Government attorney should be present as an added protection to the Government and to all participants. I am not here to prevent the full and free exchange of information. That would defeat the purpose of the Seminar. The primary reason for my presence is to guard against the inadvertent consideration of any subject which might bring the Seminar within some aspect of the antitrust laws. This is not likely in view of the rules under which the Seminar is held and the excellent manner in which these Seminars are always conducted. The agenda has been prepared with a view of permitting free discussion of the important topics to be considered. I will be present through all the sessions. If at any time I think we are straying into an area which might raise antitrust implications, I will call this to the Chairman's attention so that any such discussion may be avoided. I will also be available at all times during the Seminar both during and outside of meetings for the consideration of antitrust, conflict of interest, or other legal problems which may arise. I should add that I enjoyed myself very much at last year's Seminar, met many interesting people, and also learned a great deal. I am very happy to be back again today.

Col. McCants: Thank you Mr. Docherty. Gentlemen, subjects placed on the agenda of this Seminar were selected for presentation for the sole purpose of progressing the science of explosives safety. Products and/or ideas presented by participants do not necessarily have the indorsement of the Department of Defense.

AN ACCIDENT IN THE PNEUMATIC CONVEYING  
OF A MISSILE PROPELLANT

---

by

J. E. Settles, Manager of Safety  
Chemical Propulsion Division  
Hercules Powder Company  
Wilmington, Delaware

Hercules Powder Company experienced an explosion at Allegany Ballistics Laboratory on Thursday, July 16, 1964. The accident occurred in the pneumatic conveying of casting powder for a more advanced propellant formula. This particular propellant formula contained a burning rate accelerator in the form of metal strips. This metalized formula was known to be sensitive to electrostatic conditions and to friction and an appreciable amount of study and testing had been directed toward characterizing the limits of this sensitivity and the factors of safety that would be existing in the system.

In view of the fact that the accident occurred, it is obvious the studies that were performed did not go far enough. Additional studies are now under way, and it must be emphasized that these additional studies are very detailed in nature. It is not possible at this time to present final and conclusive answers. This is very much of an interim report.

The objective of this discussion at this time is to point out some of the problems involved in designing a safe pneumatic conveying system for propellants and explosives. The published agenda did not list this paper. In discussing the matter with Colonel McCants of the ASESB after the ABL accident, it was agreed that the propellant industry may be able to profit from these experiences in a very material way, particularly if any one now present uses pneumatic conveying systems for moving propellants and explosives or if any one is considering such a possibility.

A first and obvious question which should be answered is: Why convey pneumatically?

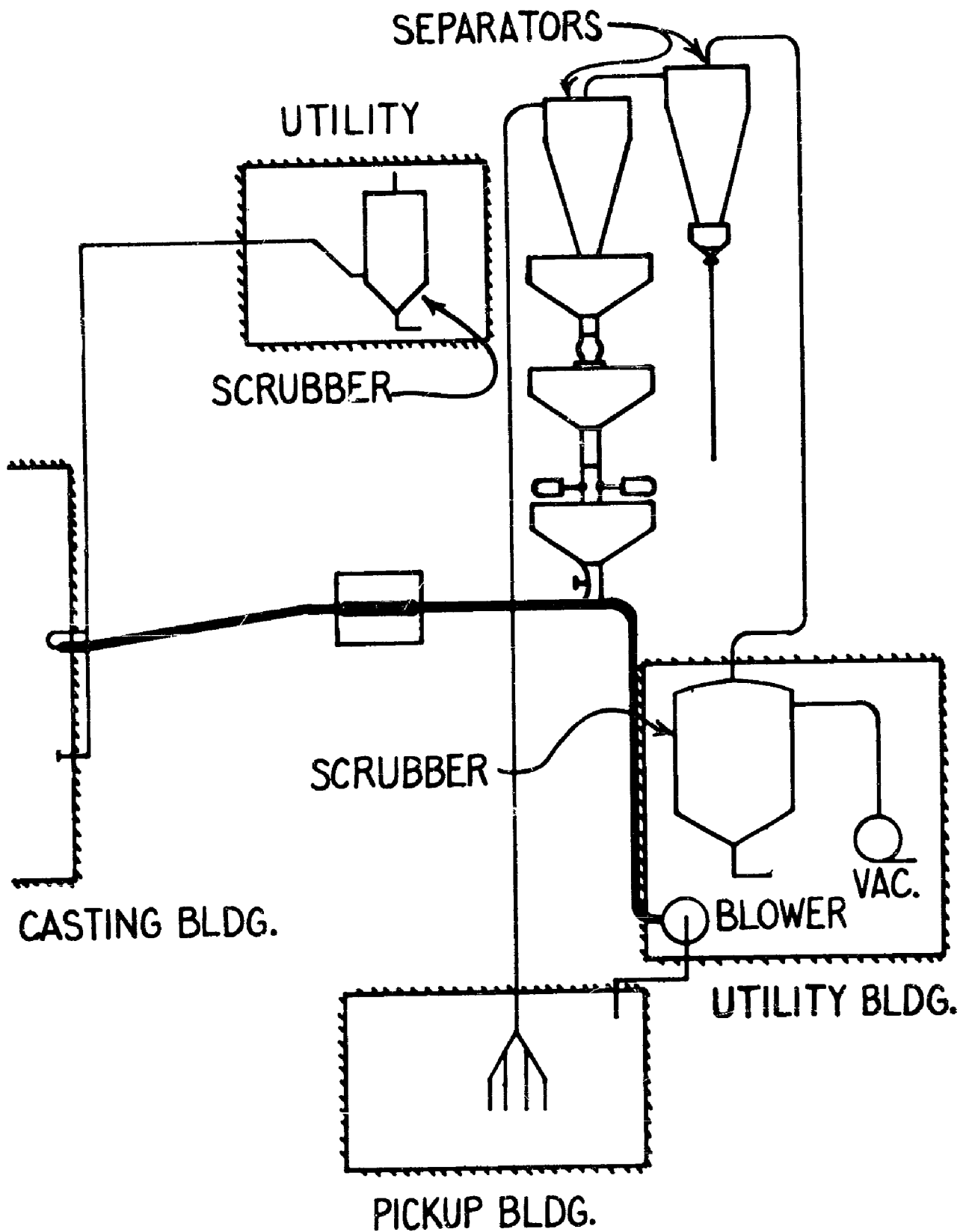
There are two very good answers for this question. One is efficiency. Mass movement of material is always more efficient. At this point it can be reasonably suggested that mass movement can also be accomplished by devices other than pneumatic conveyors. There are, for instance, mechanical conveyors such as belt conveyors and also vibrating conveyors which will move materials very efficiently and effectively. However, long systems of these designs are expensive to install and at this point the second answer to

the question of "why convey pneumatically" becomes very important. It is more desirable to move propellants and explosives pneumatically because of the added safety factors. In a well-designed pneumatic conveying system there will be no metallic working parts to become contaminated with sensitive ingredients and possibly cause an accident. The potential for a mechanical failure to occur which would result in an accident is much reduced in a pneumatic system; also, control of hazardous dust conditions is much easier but most importantly pneumatic conveying systems make it possible to eliminate the exposure of people to potentially hazardous conditions during the loading, transportation, and unloading of granular propellants and explosives.

However, pneumatic conveying does have problems. One of these problems is the heat of friction which results from the high speed travel of granular materials through a conveying pipe. Another problem is electrostatics. It is strongly indicated the potential safety advantages in a pneumatic conveying system make it well worth while trying to find solutions to these two problem areas.

The details of the accident which occurred at ABL on July 16 can be better explained with the following slide. This is a schematic of the pneumatic conveying system which was being used.

# SYSTEM LAYOUT



The major components of the ABL system included: (1) a powder pick-up station which was located in a separate uncovered building; (2) a pneumatic conveying system--which utilized vacuum to move the propellant into an interstage tower; (3) the tower which was followed by the last component, (4) a pneumatic conveying system which utilized pressure to move the propellant into a hopper in a Casting Building. In viewing the system the obvious questions are: Why was a combination of a vacuum system and a pressure system being used and what was the purpose of the interstage tower.

By using vacuum it is possible to efficiently, effectively and safely transfer the granular propellant from the original shipping containers, which are usually boxes or drums, containing 150 lbs., into the conveying system. The pressure system was desirable for the final movement of the powder into the casting building because of the length of the system. At distances of 200 to 500 feet, it is more economical and efficient to blow the powder rather than to attempt to suck it through the system. The interstage tower served two purposes. First, it was a necessity in order to transfer the material from a vacuum system to a pressure system. Secondly, it served as an interruption in the stream of propellant between two operating buildings. As a matter of policy and precaution it is never desirable to tie two buildings together with a continuous stream of explosives. If two buildings are connected with a continuous stream of explosives, an accident in one operation is almost certain to propagate to the adjacent operation.

An eye-witness of the ABL accident established as factual evidence that the first explosion occurred in the Casting Building. A few seconds later a second explosion occurred at the interstage tower. The distance between these two points was approximately 225 feet and the conveying air velocity was approximately 9,000 feet per minute.

The two and one-half inch I. D. pipe which was used in the conveying system was buried under ground to protect it from external forces and for temperature control. Several small explosions occurred in this pipe: the first about 40 feet downstream from the interstage tower, and then at about twenty or thirty-foot intervals as an investigator would move toward the Casting Building. With only one exception, the pipe itself was not ruptured by these small explosions. The pressure buildup was relieved at the points where flexible couplings held the pipe sections together.

No explosion occurred in the powder pick-up building. This structure was essentially undamaged. After the explosion the four containers in this building from which the powder was being drawn contained an average of about 8 pounds of powder, so the boxes were essentially empty.

The interstage tower was completely destroyed by the explosion. This tower consisted of a tangential separator at the top, with three separate hoppers below it. During operations, powder flowed continuously from the tangential separator into the first hopper. At pre-determined intervals a plug valve--the operation of which was external to the system--located at the bottom of the first hopper would open and permit the next lower hopper

to fill. After the plug valve between the first and second hoppers closed, a pneumatically operated pinch valve between the second and third hoppers would open and permit the powder to flow into the lower hopper. Flow from the lower hopper into the pressure conveying system was continuous through a throttled valve. The valves above and below the middle hopper were never open simultaneously.

It is estimated that there may have been 60 to 70 pounds of powder in the interstage tower.

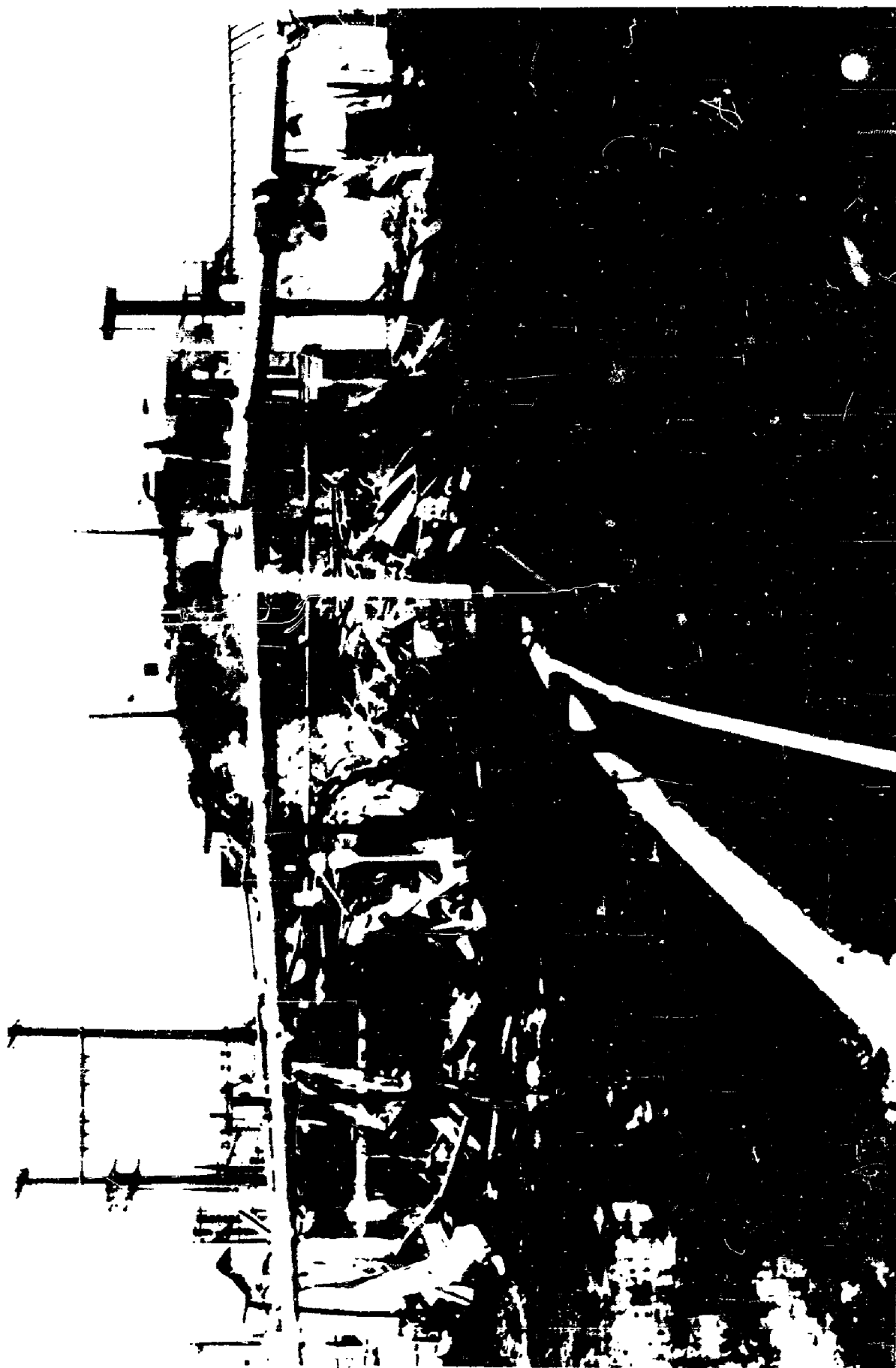
All operating personnel were housed in an earth-covered remote control shelter and were uninjured by the explosion.

Although the first explosive reaction which was visible externally was in the Casting Building, the initiation point could have been in the conveying system. The evidence indicated that if such was not the case then initiation probably occurred in the receiving hopper in the Casting Building. An explanation for this latter possibility might be that--although this entire system was exceedingly well grounded from end to end because of the electrostatic potential--the free-fall of powder granules through the interior of receiving hopper--a distance that could have been six or eight feet--could have resulted in an electrostatic buildup and discharge which caused an ignition of the dusty atmosphere within this hopper.

If ignition occurred within the conveying system, it is almost certain to have been the result of frictional contact between the powder granules and the walls of the conveying pipe. The friction sensitivity of the particular powder formula being conveyed had been carefully checked out by ABL engineers in a pilot scale pneumatic conveying system. The data which was obtained in these tests strongly supported the conclusion that the relationship between the velocity of the conveying air and the velocity of the solid particle being moved by the air would not result in initiation by friction.

The following two pictures give an external view of the Casting Building after the explosion and the spot at which the interstage tower stood. The tower was completely destroyed.







Certain considerations involved in the ABL accident are of interest. These can be better explained if there is a brief review of what was known about pneumatic conveying before the accident.

Some early experiments into the fluidization of solids were performed by W. K. Lewis, E. R. Gilliland, and W. C. Bauer and were reported in a paper published in Industry and Engineering Chemistry in 1949. These authors in studying the effects of particle size and density upon the air velocity necessary to fluidize solid particles concluded that the difference between the carrying air velocity and the particle velocity is about the free-fall velocity of the solids in still air; thus, if the air velocity is known, the particle velocity may be calculated by subtracting the free-fall velocity from the air velocity. If the free-fall concept is valid, it can be computed that the particle velocity will very nearly equal the velocity of the conveying air at the higher rates of flow, i. e. around 10,000 feet per minute.

However, at the time Lewis, Gilliland, and Bauer published their studies, the Dallavalle method of correlating air velocity and particle velocity was available to the industry. In 1942, J. M. Dallavalle published this method, and it indicated the free-fall velocity (or terminal velocity) is only a measure of the minimum velocity necessary to just fluidize a mass of solid particles.

Transporting solids by higher air velocity was the subject of papers published by Dr. B. L. Hinkle of Georgia Institute of Technology in 1953 and by Dr. C. Y. Wen at West Virginia University in 1956. Some of the findings of these authors are as follows:

1. The solid Velocity was independent of the solids loading.
2. The rate of acceleration of solids to their equilibrium velocity is inversely proportioned to the solids loading.
3. The particle velocity is approximately equal to one-half of the air velocity.

Dr. Hinkle's experiments cover the following set of conditions:

Air Velocity	- 66 to 118 ft/sec
Particle Diameter	- 0.014 to 0.33 inches
Particle Density	- 66.5 to 115 lbs/ft <sup>3</sup>
Tube Diameter	- 2" and 3"

Dr. Wen found the solids velocity to be about one half the gas velocity. He also presented a correlation relating solids linear velocity to solids mass velocity, solids density and tube diameter.

Recently our engineers at ABL contacted Dr. H. P. Simons, Head of the Department of Chemical Engineering at West Virginia University. Dr. Simons indicated that to the best of his knowledge the slip velocity is inversely proportional to the density of the material. Dr. Simons also indicated that data published prior to about 1955 or 1956 concerning particle transport mechanisms is somewhat "suspect" in light of the latest experiments now being performed at West Virginia University. Data from these latest tests at West Virginia University are not available at this time.

Prior to the accident, a series of tests were conducted at ABL in pilot-phase type of equipment. The particular formula of propellant which was involved in the accident was included in this series of tests. The maximum granule velocity which was recorded in this series of tests was approximately one half the velocity of the conveying air. At an air velocity of between 9,000 and 10,000 feet per minute, with a corresponding granule velocity of less than 5,000 feet per minute, no evidence of decomposition or ignition was observed.

It was established in this series of tests that if a particle velocity of between 9,000 and 10,000 feet per minute were to occur that frictional initiation of this particular metalized formula might occur. However, since the velocity relationships which were observed in the tests correlated with the more recent opinions published in the literature, they were considered valid. On the basis of such available data an initial production run was scheduled for the particular metalized formula of interest. The accident occurred during that first production run.

Investigation of the pneumatic conveying of granular propellants was started at Radford Army Ammunition Plant in 1956 and an installation was made in the mold filling operation at that plant in 1959. A comprehensive pilot phase investigation of the conditions which would exist in a pneumatic conveying system was conducted at the Radford plant during this period of time. The testing equipment utilized included parallel conveying pipes 100 feet in length. The pipes were 3 1/2 inches in diameter.

One pipe was constructed of steel and the other of transparent plastic so that physical observations of flow characteristics could be made. Conventional cannon powders and single and double-base casting powders were tested in this equipment. Conveying air velocities of up to 10,000 feet per minute were investigated, and it was determined that corresponding particle velocity was a function of granule size, density, and rate of loading. Within the limits of these variables, particle velocities were observed to range from 5,000 to 7,000 feet per minute when propelled by an air conveying velocity of 10,000 feet per minute. No ignition or decomposition of any of the thirteen formulas tested were observed.

Almost concurrent with the time ABL was testing in their pilot scale equipment the particular metalized formula involved in the explosion, Radford was also conducting tests on the same formula. However, in their system 100 feet long, it was possible to measure propellant and granule

velocities at more extended intervals than ABL could do with their shorter equipment. Photo-cell devices were placed in the Radford system at distances of 10 feet, 30 feet, and 83 feet, from the point at which the powder was introduced. Physical observations of conditions of flow in the transparent plastic pipe revealed an apparent phenomenon for which no explanation has been developed to date. It was observed that a bunching-effect of the powder granules took place. It seemed that the "following" granules were overtaking the "leading" granules. It was also noted that the air velocity-particle velocity relationship at the 10-foot distance and at the 30-foot distance did not correspond with the relationship which was observed at the 83-foot distance. At the longer conveying distance the granule velocities were approaching to within 80% to 90% of the air velocities. Since this data was anomalous with past data generated both at Radford and at ABL, equipment and instrumentation inaccuracies were suspected. During the period of time these discrepancies were being investigated, the accident occurred at ABL.

It has been indicated that this is an interim report. As of now, the exact cause of the accident at ABL is not known. It may never be possible to arrive at an accurate conclusion. The problems involved in pneumatic conveying of propellants and explosives are still being intensively studied. Other papers on this subject are being given at this seminar, and they may answer some of the questions which are left unanswered by this paper.

However, several precautionary considerations can be drawn from the circumstances involved in the ABL accident. The precautions are:

No. 1 - The research and testing which has been conducted by others is important as background information. However, such data should not be used as design criteria without verification and checking. The only data exactly applicable to your particular problem will be data involving your particular system and your particular formulas.

No. 2 - The relationship between the velocity of the conveying air and the velocity of the granular solids which are being moved must be accurately established under the exact conditions which will exist in the system that is to be used--if the solids being moved are friction-sensitive.

No. 3 - Some estimate should be made of the Reynolds number of turbulent areas within your system and an evaluation should be made of the effects of this turbulence on the conveying characteristics and hazards of your system.

And associated with the foregoing:--special attention should be given to the radius of the bends and tube-turns within your system. Such areas are of particular importance if the formula being moved tends to be friction-sensitive.

No. 4 - It should not be positively assumed that a well-grounded system adequately takes care of the electrostatic problem.

No. 5 - Every meaningful formula change should be separately evaluated.

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Mr. King: What was the pipe diameter?

Mr. Settles: 2 1/2".

Mr. King: Was the internal atmosphere of the pipe controlled?

Mr. Settles: Yes, it was controlled.

Mr. King: What was the relative humidity?

Mr. Settles: It was around 50% I believe.

Mr. King: Did you have any electrostatic probes inside of the pipe?

Mr. Settles: Not for this particular set-up.

Mr. King: Could this incident have been associated with the pick-up of the powder?

Mr. Settles: I don't know Paul, we don't know why it happened, we haven't been able to develop a reason as yet.

Mr. Wiuff: Did you evaluate your test using inert or nitrogen gas?

Mr. Settles: No we did not.

## THE PROGRAM OF THE ARMED SERVICES EXPLOSIVES SAFETY BOARD

Russel G. Perkins, ASESB

The Chairman and Secretariat thought that (since you are captive and have to listen) the group might appreciate a few brief comments on the activities of the Board since the last Seminar. We want you to feel that we have been busy in your behalf during the year and that certain tentative promises made a year ago have been fulfilled. Some of the items we are reporting will be related to presentations later during this year's Seminar and may serve to answer questions you have in your minds. Some of the topics will bring subjects to you that are relatively new.

Board safety survey functions are responsible world-wide for all Department of Defense facilities handling ammunition and explosives. They have paid specific attention during the last year to those areas or conditions wherein if an incident were to occur, it might become of international significance because of heavy risks of deaths or injuries among the local populace. Not only do some situations of this gravity exist overseas, but (without the international complications) a few conditions of this severity also exist within CONUS. After a careful study from both a safety and operational viewpoint, the Board deemed it appropriate in a limited number of cases to advise the Secretary of the Military Department concerned and the Secretary of Defense of the existing conditions. For the majority, satisfactory resolution could be obtained at the local level. In a few cases the recommendations suggested were given serious consideration by higher headquarters and quite frequently suggestions were adopted to eliminate the hazard or reduce it to an acceptable degree. The implementation of the Board's recommendations in eliminating many hazardous conditions has brought great satisfaction to the Board and its personnel.

Work on the revision of ammunition hazard classes has been completed resulting in the issuance of DOD Instruction 4145.22 dated 18 May 1964 and entitled: "Quantity Limitations and Quantity-Distance Standards for Manufacturing, Handling and Storing Ammunition and Explosives." Essentially, this document establishes eight new hazard classes for ammunition and explosives in place of the previous twelve classes. This is the first opportunity in approximately 35 years for us to have an up-to-date classification system that is abreast of the manifold recent changes in weapons technology. Classes 1 and 2 remain essentially the same; Classes 3 through 6 establish various distances from 400 feet to 1800 feet to protect

against increasing ranges of fragments from the more hazardous types of ammunition. Class 7 includes mass-detonating material and uses the quantity-distance tables applied to the former Classes 9 and 10. This Instruction, at present, is being implemented by the Military Departments.

Work Groups on Explosive Equivalency of, and Storage Criteria for, Liquid Propellants completed their missions with the issuance of DOD Instruction 4145.21 dated 26 March 1964 which is entitled: "Quantity-Distance Storage Criteria for Liquid Propellants." This criteria includes the storage of both fuels and oxidizers in bulk quantities and assigns compatibility groupings and explosive equivalencies for mixtures of fuels and oxidizers. These criteria are based upon extremely limited data. The urgent requirement that criteria be developed in this area dictated that this action be taken in advance of the acquisition of the needed data. These work groups have not been disbanded as it is anticipated that they will provide overall consultant services to the various continuing efforts to refine this criteria.

One such effort designated "Project Pyro" is a program of explosion tests at the Edwards Air Force Base. These are jointly funded by the Department of the Air Force and the National Aeronautics & Space Administration. They are fully instrumented tests to determine peak pressure and impulse for determination of "high explosive yield" from various combinations of fuels and oxidizers.

Our Work Group to Develop Minimum Test Criteria for Ammunition and Explosives has held a number of meetings during the past year in an effort to revise the existing tri-Service document in order to include more realistic tests for large boosters, and to include up-to-date, meaningful tests particularly with respect to standardization of instrumentation. It is estimated that the revised document will be published by approximately 1 January 1965.

In numerous discussions around the country, the term "high explosive equivalent" or "high explosive yield" has been bandied about a great deal. Industry, the Military Departments, and the Space Agency all have a vital need for further definitive information on the validity of this concept. Some of the comments on this subject indicate that it is a rather sore point.

Included in the problem are the important questions as to (1) whether or not simple increase in the overall amount of material involved in an incident, and (2) confinement or lack of



it would serve to increase the so-called "high explosive yield" percentage figure.

Some of the work done in "explosives safety investigations" on dividing walls has, for example, indicated a need for further work on the behavior of combinations of high explosives and non-detonable propellants in storage situations which provide moderate to complete confinement. The effects of systems which contain both a high explosive portion and a non-detonable propellant portion are sometimes described or measured by a so-called high explosive equivalency figure for the total quantity of explosives, including the propellant, at a percentage of its actual weight. There is some indication, at least, that the high explosive yield figure obtained with even moderate confinement would be somewhat higher than with the charge situated in free air.

We have previously suggested our suspicion that the "high explosive yield figures" may vary depending upon the total amount of explosive involved in the incident and the distances from the charge at which pressure and impulse measurements are made.

In order to obtain a start on a solution to these problems, the Board recently authorized a series of tests at the Naval Ordnance Test Station, to improve our understanding of the behavior of propellants involved in explosive incidents particularly in complete systems when a portion of the explosive charge is known to be a detonable propellant or is a high explosive warhead of considerable size. Initial phases of this program are funded by NASA with a voluntary contribution to our explosive safety evaluations. I would be remiss if I did not acknowledge here the most welcome assistance of Doug McCauley and Norm Peil (who unfortunately could not be with us), and Erskine Harton in supporting and gaining approval for this. What is envisioned is a program in which only three variables would be allowed among several tests to determine for a particular combination of Class 2 and Class 7 propellants if high explosive yield figures vary.

A series of tests would be run with a suitable ratio of the two items - say, 50% of each with successive tests being of larger amounts; for example, 16,000; 32,000; and 64,000 pounds to determine if simple increase in the size of the incident would lead to increases in the size of the computed high explosive yield.

In conjunction with this, a series would be run in which the charges would consist of the same items but the ratio of Class 7 to Class 2 would be varied in separate tests such as 25% to 75% of the mass-detonating component.

If a significant "high explosive yield" figure is determined for one of the combinations in an open-air shot, it is then planned that an identical test would be performed with the charges located in an earth-covered igloo. This would enable us to determine how much, if any, increase in the so-called high explosive yield of the charge involved would occur as a result of the confinement afforded by the earth-covered structure.

In all of the tests proposed there would be sufficient redundancy in instrumentation to assure valid results over a considerable range of distance. Also, specific tests, where pertinent, would be repeated in exact duplicate to get a larger amount of the appropriate data and provide a more sound basis for the "high explosive yield" figure derived.

Keep in mind that the purpose of the proposed program is to provide a measuring device or calibration standard for future work by yourselves and others in determining the proper hazard classification and quantity-distance characteristics of the weapon systems you develop.

It is our hope that a very limited number of tests will suffice to provide a basis for enabling different operating organizations to determine yield figures which can be safely used for evaluation of different systems and so that the results obtained for each of the different systems will be truly correlatable with those of others.

Included in this program will be careful observation of the dispersal of fragments and burning debris from the tests. Such studies as these are included in the destructive tests of structures, as well as Board Staff studies of incidents and the test work carried out by other organizations. It is hoped that the data derived therefrom will provide a sound and useful basis for evaluating some of the incomplete and uncertain points in our existing DOD quantity-distance instructions and criteria.

As in our other tests we plan to make use of surplus explosives to reduce the overall costs. At this time we have only a limited number of motors in sight so we earnestly enjoin any of you who have appropriate programs to be on the lookout for large motors which might be suitable for this purpose.

At past seminars, you have heard some of the results of our dividing wall studies. Last year we promised a film report on this. Later on in this seminar we will present a color sound documentary film which has been compiled to give you a really good overall look at the entire program. It appears that we can get approval for

unqualified release so that this can be made available not only to the Military, but schools and libraries.

The original concept of the dividing wall test program was to investigate the efficacy of barrier walls in prevention of the propagation of explosions. Some slight broadening of this limited field has been necessary because of a critical need for information in related fields. The general program has been tentatively assured funding of \$880,000 per annum for the next two years and the basic investigations will continue.

Some months ago the Board determined that uncertainties in the application of barricaded distances existed, particularly for such situations as intraline separations for multi-story buildings.

Engineers and technicians associated with the analysis and design of barricades to provide facilities protection from the efforts of explosions have expressed increasing concern relative to the effectiveness of barricades as a positive method of protection. Limited scientific data, including high speed motion picture sequences and test instrumentation, have indicated that a lesser degree of protection may be afforded by the barricades than has been assumed in the past. If, in fact, barricades are not adequately serving the intended purpose, adjacent public properties and on-base facilities may be exposed to serious damage and loss of life should an incident occur.

In order to fully resolve all questions pertaining to the problem of barricading, consideration must be given to the following aspects of the barricade design:

- a. Effectiveness in attenuating blast and fragment effects.
- b. Optimum location in relation to donor and target.
- c. Most effective cross-sectional configuration and economical construction.
- d. Based upon the present state of knowledge, the height of a barricade is usually determined by the height of the structure to be protected and generally speaking, the cross-section is the same whether the hazard is 500 pounds or 500,000 pounds.

Test work may well be needed to answer specific questions in the area of barricading, but prior to undertaking any such work, the Board formally recommended a thorough search of available literature on the subject to provide a sound working basis for directing such tests. After exhaustive investigation of a number

of means suggested by the Board Chairman, the Assistant Secretary of Defense (I&L), working in conjunction with the Chairman, made the decision that all things considered a requirement existed for the assignment of a full-time team to this study for a suitable period of time. The Secretary took steps accordingly. Specifically, he requested that the three Military Departments each assign a qualified individual to the Board for an uninterrupted period approximating ninety days for the purpose of forming a team to execute this study. It is anticipated that our most worthy and respected consultant, Mr. Henry Marsh will continue to render a most valuable service to us in this regard.

Since the last seminar the Board Secretariat has secured an additional engineer to assist in the review of plans for the construction of explosives facilities to provide for even more expeditious service in this vital area of the Board's responsibilities. In addition, another work group is nearing completion of the revision of the former DOD Instruction 4500.12 "Special Instructions for Drivers of Motor Vehicles Transporting Explosives and Certain Other Dangerous Articles." The primary purpose of this is to provide the driver with specific instructions as to what he should do in the event of an accident and to provide information for the use of firefighters and police as to the hazards involved with the cargo. There is a serious deficiency in this system which we recognize. It is that the driver is the sole possessor of the information. The firemen and police must contact the driver in order to obtain this information. We are convinced that the answer lies in developing a uniform placarding system which will give instant visual recognition of the hazards involved with the cargo.

Since the ICC has the responsibility for regulating the transportation of explosives and other dangerous articles, the Chairman and Secretariat intend to move in the near future to explore the possibility of the ICC joining with the Board in developing such a system which would be subsequently incorporated in the ICC Regulations. The Board is convinced that it can perform a real public service in this area. You may desire to interest yourselves in our progress.

This has been a very rough and quick review of several areas of the major effort being put forth by the Board. If you have questions or comments regarding any of the subjects which I have touched upon, please feel free to bring them up at appropriate times during the formal sessions of the seminar or informally as you have an opportunity.

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## THE IMPACT OF EXPLOSIVE EQUIVALENTS ON LAUNCH SITE OPERATIONS PLANNING

W. G. Hardy  
General Dynamics/Astronautics  
San Diego, Calif.

Let me clarify at the outset that the following does not pertain to operations planning in the sense of deployed operational military missiles, but rather to development test activities of launch and/or ground test stands and space vehicle launching sites.

Facilities and operations planning is very much like every other engineering problem, one has a given set of inputs, required outputs, and constraints. It is necessary to examine various solutions that will satisfy the given information, using as trade-off criteria cost, performance, and schedule. The problem is complete when the final selection can be expressed in quantitative terms such that the outputs are visible functions of the inputs and constraints. It should be mentioned that when speaking of performance we include such things as reliability, maintainability, and safety as well as other measurable characteristics of a system and how well it performs.

The inputs are generally expressed as operational requirements, included in these are the orbit parameters of altitude and inclination which may be transformed into launch parameters of direction and geographical location using the known vehicle capabilities. Also known are the launch rates and the schedule requirements. Of importance is the vehicle descriptive information of size, propellant type and quantity, and thrust level. The constraints may consist of such things as existing sites and support facilities, including down range tracking, recovery, etc. Site hazard criteria may be given for conditions within the site and limitations relative to civilian personnel near the site. Of course physical constraints must be considered, such things as the number of people who can work in a given area at one time, transportation and handling capabilities, and the fact that only 24 hours exist in a day. The outputs will be such things as site layouts, equipment design requirements, facility design requirements, schedules, manpower requirements, and safety requirements.

Let us examine for a moment the launch siting problem and factors that affect it. The initial planning may be done without regard to existing real estate. This might determine the optimum

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or ideal case which can later be modified as required to fit a given location and its real estate boundaries. Consideration must be given to the launch rates, vehicle propellant information, such as whether cryogenic, toxic, etc., and thrust levels. The locations of the launching or test stands, storage vessels and support areas must be defined. Because for liquid propellant vehicles both propellants will be utilized on the test stand vs only one at a time in the storage area, the separation distances of prime concern are stand to stand, stand to support areas, and stand to uncontrolled areas. Similarly for solid propellant vehicles because for very large vehicles the motors are segmented or clustered. Factors to be considered are blast overpressure limits, sound pressure levels, overflight restrictions, set-back requirements, prevailing winds, and toxicity of exhaust products or boil-off gases. If the launch vehicle is manned, consideration must be given to abort situations, considering such things as reaction time and detection capabilities, the type of hazard, whether fire or detonation, and clear areas for the impact of personnel.

Blast pressures are determined from the quantity of explosive involved. Because launch vehicles use various combinations of fuels and oxidizers, it is convenient to convert the propellant quantity to some standard quantity of explosive. The quantity of propellant is multiplied by an explosive equivalent factor and the result is normally expressed in pounds of TNT.

Unfortunately, there is a general attempt to oversimplify this equivalent in the form of a constant percentage. For planning of future vehicles this is often necessary because of many unknowns. However, it is very important to keep in mind that TNT equivalent is not an intrinsic property of the propellant combination but is a function of the propellant combination modified by vehicle geometry, that is critical diameter and length to diameter relationships.

Let's take a look at the quantity of propellants we are talking about. The NASA Saturn V launch vehicle currently under development has, according to the published literature, a first stage containing 4.4 million pounds of LOX/RP-1 propellants. It has a second and third stage totaling 1.2 million pounds of LOX/LH<sub>2</sub> propellants. Looking into the future effort, large vehicles called the NOVA class, have been studied which contain approximately 25 million pounds of liquid propellants or 30 million pounds of solid propellants in combination with liquids. In the latter case, the solids account for most of the weight.

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What does this mean in terms of explosive equivalents? An examination of the theoretical energy in the LOX/RP-1 combination shows a greater than 100% TNT equivalent. Does this mean that we must plan for yields in the 10 to 15 kiloton range. Fortunately not, because the mixing of propellants prior to detonation will be much less than 100% and there will be a great deal of burning, particularly if the propellants are hypergolic. Also of importance is the geometry of the vehicle and the arrangement of the oxidizer and fuel tanks which will affect the mixing of propellants in the event of an incident. The yield is also a function of the impact sensitivity, that is should the vehicle topple before ignition or after a fire has started or fall back because of malfunction early in flight.

Scale tests have been conducted of the LOX/RP-1 combination in which it was determined that the TNT yield is much less than 100%. In addition, there have been incidents involving full scale vehicles which confirm the results of the scale test, at least for certain vehicle configurations. For purposes of future planning, the TNT equivalent of this combination is based upon tests to date and experience with present day vehicles.

Figure 1 illustrates the relationship of separation distance as a function of equivalent pounds of TNT for several overpressures. This is expressed by the equation  $d = ze^{1/3}$  where  $z$  is a function of the overpressure and  $e$  is the quantity of TNT equivalent. Overpressures of 0.2 psi are normally allowed for uncontrolled areas and 0.4 psi for adjacent launch installations which are not occupied during those times when the vehicle is tanked with both propellants. Also shown is a curve for Class 9 and 10 explosive storage which has been extrapolated to very large quantities. Note that this curve is equivalent to approximately 0.5 psi. It might also be mentioned that overpressures created by hurricane velocity winds are between 0.25 and 0.3 psi.

Figure 2 shows the relationship between separation distances and psi overpressure for a one million pound explosive equivalent. As can be seen, the curve rises sharply below 0.25. Figure 3 shows the relationship between distance and explosive equivalent. Note that if very high TNT equivalents are used, that is between 80 and 100%, the distance requirement is quite insensitive to the percent equivalent. However, for values much less than 50% TNT equivalents, the distance can increase markedly.

For a given propellant quantity, then, the only way to reduce separation distances, assuming this is desirable, is to either accept higher overpressures or determine that the percent of TNT

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$$d = ZE^{1/3}$$

# SEPARATION DISTANCE

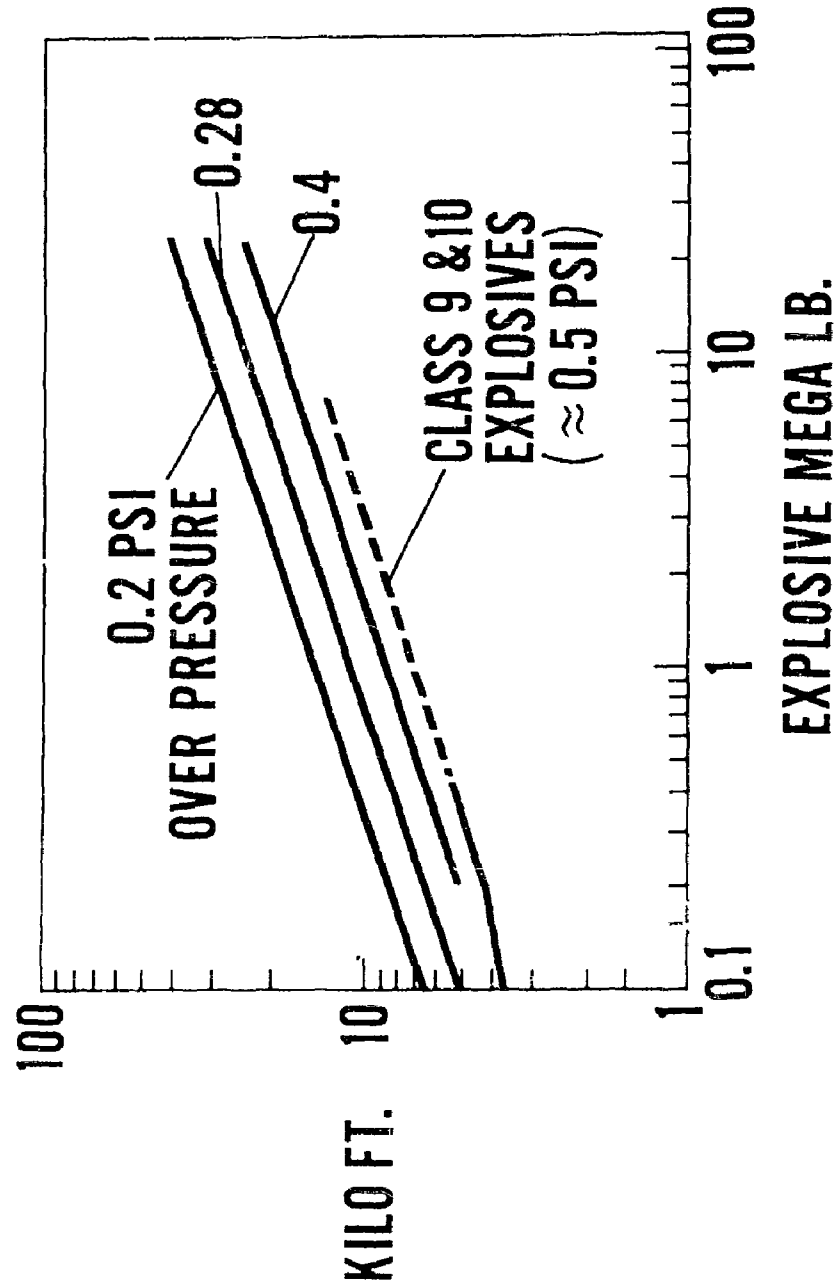


FIGURE 1

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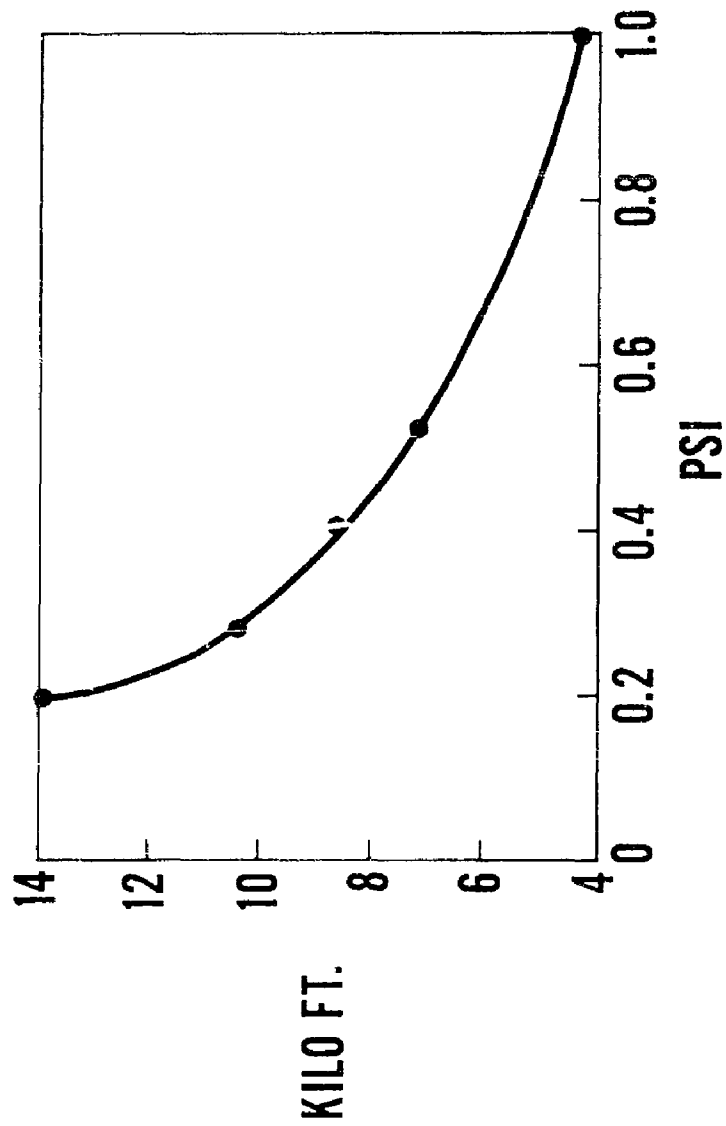


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# SEPARATION DISTANCE vs OVER PRESSURE

$$d = ZE^{1/3}$$

1 MEGA LB.

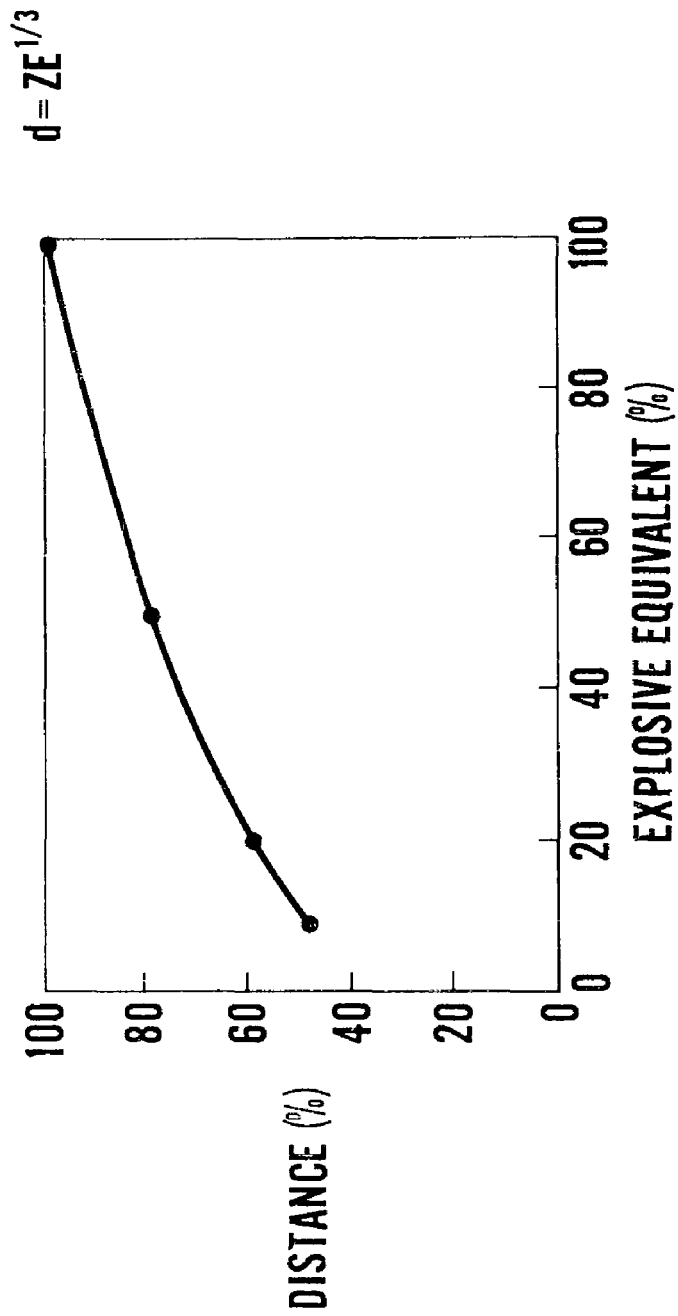


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FIGURE 2

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SEPARATION DISTANCE vs % EXPLOSIVE EQUIVALENT



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FIGURE 3

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equivalent is less than initially assumed. In the latter case, tests and analytical studies are required to establish or adjust the values.

There is one other approach which suggests itself and that is instead of limiting the potential overpressure to a "safe" value for uncontrolled areas, allow it to be greater and determine the probability of occurrence, i.e., take a calculated risk. This may sound all right if one rationalizes that the probability is sufficiently small. This is not recommended, however, I recall an incident that occurred a few years ago at the Eastern Test Range when a solid propellant vehicle exploded on the pad. A piece traveled a considerable distance and damaged a vehicle on another stand. The chances of this seem so remote that prior to the incident many people took exception to the safety ruling that required the vacating of people from the stand that was struck. Needless to say, after the incident, the arguments were fewer.

As mentioned previously, the LOX/RP-1 propellant combination is quite well known as it should be because of its use for the past several years in missiles such as the Thor and Atlas. Other combinations are not as well known such as LOX and LH<sub>2</sub>. In these cases relatively high explosive equivalents are usually assigned with the resultant increase in costs.

Solid propellants present similar problems. Single base propellants are usually treated as Class II explosives which may be expressed as a very low TNT equivalent and signify that they are primarily a fire hazard. There remain other areas of unknowns, however, and that is the sensitivity of a given solid rocket to impact. For instance, suppose a malfunction should occur on lift-off which would cause the vehicle to fall back in the vicinity of the launch site. Under these conditions, an explosion may be possible even with a so-called Class II propellant.

The application of solid rockets in certain vehicles such as the Titan III, currently under development by the Air Force, presents a new realm of possibilities. In this case the solid propellant motors are attached to a liquid fueled rocket. In the event of an incident which ruptures the tank, what would be the effect upon the solid propellant when burning, when not burning, and with regard to impact sensitivity?

Of course most of these questions can only be answered by conducting tests. These tests must represent the particular configuration under study. The test results will determine appropriate and realistic TNT equivalents which can be used by the site

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planners. Often in the absence of complete data, plans for future systems must be made using best estimates. In order to be "conservative" these are usually on the high side.

Earlier it was mentioned that initial planning can be done without regard to actual location and then modified to fit a particular set of real estate constraints. It should be emphasized that these are very real constraints and the sooner they are applied to the problem, the better. The nation has a lot of money invested in such things as down-range tracking and recovery facilities which must be utilized. In addition there are other supporting activities near present launching sites covering the entire scope of logistics which if not utilized must be duplicated. This of course penalizes a new installation quite heavily.

Figure 4 shows a hypothetical situation of a roughly triangular shaped piece of real estate adjacent to existing launch sites and support facilities. It is bounded on one side, in the direction of launch, by the ocean and on the third side by civilian or uncontrolled area. The area available for launch pads is bounded by three lines. One is a set-back from the ocean which is a small distance independent of thrust level or propellant type. A second is an overflight limit which is also a small distance and prevents flights over adjacent work areas and facilities. The third line is the variable. Shown are two boundaries for two vehicles of identical payload capability but fueled by different propellants. This boundary is set by the overpressure which may be safely imposed upon the uncontrolled or civilian area in the event of an explosion on the launch stand. The distance from the uncontrolled area to this boundary is the separation distance taken from Figure 1 for the TNT equivalent of the particular vehicle. As can be seen, the available area is significantly affected by the explosive equivalent of the propellant.

The area between the real estate boundary and the launch pad boundary may be used for support activities because the buildings will be designed to withstand higher overpressures than civilian structures.

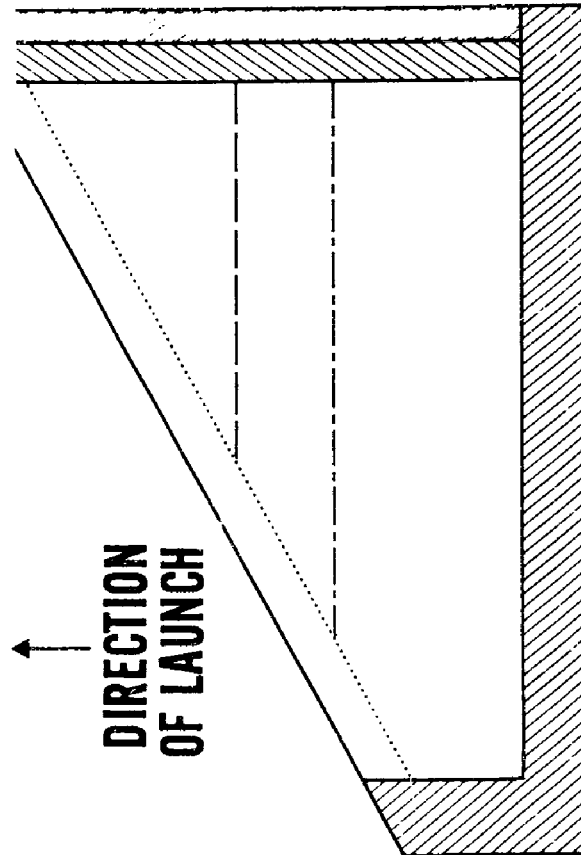
The number of launch pads that can be fitted into the available area is determined by the required pad-to-pad spacing. Thus for the vehicle with the higher TNT equivalent yield, the pad spacing is the larger and must be fitted into a smaller area. Therefore, fewer launch positions may be used. The operational requirements of launch rate and time to accomplish a launch must be examined to determine if the number of positions available will meet the requirements. Obviously, all requirements must be satisfied by

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## LAUNCH PAD AREA LIMITS

- ..... SETBACK
- HIGH EQUIVALENT PROPELLANT
- LOW EQUIVALENT PROPELLANT
- [diagonal lines] OVER FLIGHT LIMIT
- [white box] ADJACENT LAUNCH OPERATIONS
- [diagonal lines] UNCONTROLLED (CIVILIAN) AREA



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FIGURE 4

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the selected system. Therefore, the vehicle, launch site, and launch concept selected must be traded off in concert. This matter of launch concept will be examined later.

Not only must the pad-to-pad spacing be considered but also the pad to support area spacing. When we consider that support buildings such as vehicle and payload checkout buildings often must be designed to withstand hurricane velocity winds, their distance from the launch pad can be determined by this criteria without any cost penalty. This presents the opportunity to do another operational trade-off, that is to select a more efficient way of doing things.

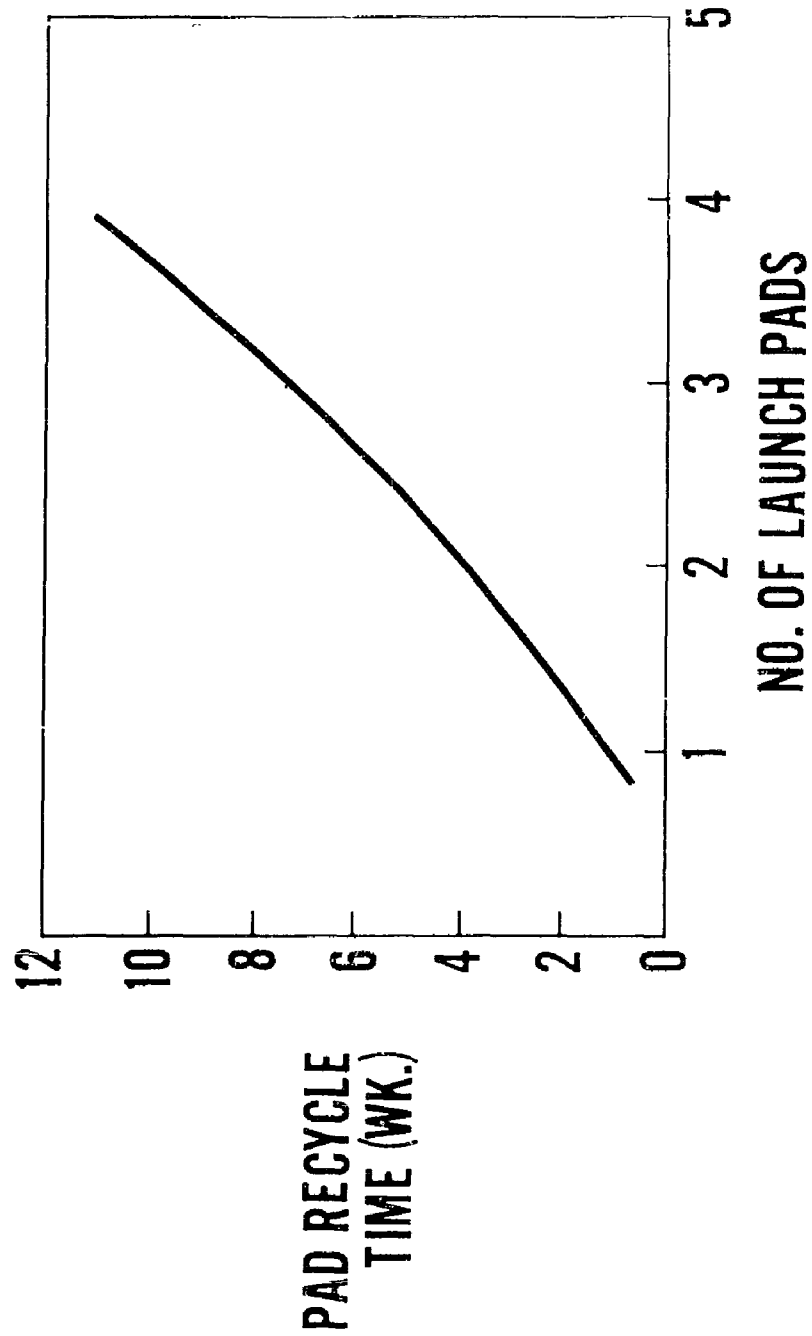
In the past, because of relatively smaller vehicles, launch pads did not require excessive spacing and real estate utilization was efficient. The launch control center was located quite close to the launch pad, within a blockhouse designed to withstand high overpressure forces. Control and instrumentation lines were relatively short and analog or direct functional signals could be used. Vehicle initial preparation and checkout was accomplished in a remote "hanger" area using similar equipment. However, upcoming and future systems require large quantities of real estate and further it is desirable from operational and cost standpoints, to use the same equipment for vehicle checkout and launch. Therefore, some newer systems incorporate the launch control functions with the other support functions. This requires much longer control and instrumentation lines which are only practical if digital techniques are used. High explosive yields was one of the functions which led to changes in operational concepts and equipment design concepts.

Figure 5 illustrates a secondary effect which may occur if existing real estate places a limit on the number of launch pads that may be installed. The pad recycle time requirement is plotted as a function of the number of pads for a constant launch rate. If the time available is very short, costs are increased and schedules are jeopardized. It may even be necessary to go to new concepts. For instance, all programs to date have used a fixed launch concept where the vehicle is assembled and checked out on the pad. However, the upcoming Saturn V and Titan III programs use a mobile concept where assembly and checkout are accomplished in one location and launch in another. The entire launch vehicle and payload are transported between sites in the vertical position, tanking and a final status check occur on the launch pad.

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**LAUNCH PAD RECYCLE TIME REQUIRED  
12 LAUNCHES PER 6 MONTHS**



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**FIGURE 5-**

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To elaborate some on abort considerations for manned vehicles which can influence site design requirements, an examination of the propellant characteristics will determine if a fire hazard or explosive hazard exists. This can be extremely important. Some propellants, those that are hypergolic, are considered to be fire hazards. If this is true, the crew may be removed from the vehicle at low altitudes or while still on the pad by use of ejection seats instead of by ejecting the entire crew capsule. This is the case in the Gemini Program for instance, in contrast to the Mercury Program. This, of course, permits a weight reduction of the payload but it also imposes a new problem for the launch site designer. Instead of a manned capsule coming down at a safe distance into the ocean, the men, with parachutes, will be landing near the launch pad. Care must, therefore, be taken to provide a sufficiently large area clear of any aboveground obstructions.

The impact of TNT explosive equivalents for rocket vehicle propellants is, therefore, seen in the real estate required, facilities costs, and the selection of launch site concepts.

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# A CRITERION FOR PREDICTING IMPACT INITIATION OF EXPLOSIVE SYSTEMS

Hyla S. Napadensky\* and J. E. Kennedy\*\*  
IIT Research Institute, Chicago, Illinois

## ABSTRACT

A sensitivity test has been devised wherein cylindrical, unconfined specimens of high explosive of the order of a pound in weight are squeezed between an explosively driven plate and a massive anvil. At one extreme of the initiation spectrum the shock transmitted is of sufficient intensity to initiate detonation before or at the instant when it reaches the face of the anvil. At the threshold of initiation, however, the time between initial movement of the plate and evidence of explosion is long enough for extensive crushing of the HE to occur. The critical impact velocity required for initiation has been found to be strongly dependent upon the geometry of the explosive specimen. Sensitivity increases rapidly as the length of the specimen is decreased. Marginal initiation is the result of a slow crushing process for short samples. As specimen length is increased, the critical impact velocity approaches a limiting upper value which is sufficient for shock initiation of the explosive. Graphs illustrate the variation of critical impact velocity with specimen geometry. High speed photographs are shown of the severe cracking and breakup of the explosive during impact.

Laboratory data obtained in this sensitivity experiment have been utilized to accurately predict the impact response of weapons containing HE. The particle velocity induced in each interior explosive element of a weapon was calculated as a function of impact velocity using stress wave propagation equations. The predicted value of the critical weapon impact velocity was that at which a critical particle velocity was induced in one of the explosive warhead elements. The critical particle velocity for each explosive warhead element was determined by the length of the element, from a graph of the experimental results. The success of the prediction method is ascribed to the similarity of the laboratory experiment to the weapon impact case in that it accounts for the long duration load and the crushing of explosives which controls weapon response.

It is believed that this experimental technique which was developed for evaluating impact sensitivity of large HE charges can be effectively applied to characterize the effects of impact loads on solid propellants initially burning or unlit in order to simulate, for example, the sequence of events following the impact of a burning rocket motor or large grain segments of a destroyed motor.

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\* Research Engineer and Group Leader, Explosion Mechanics, IIT Research Institute, Chicago, Illinois.

\*\* Associate Research Engineer, Ballistics and Explosives, IIT Research Institute, Chicago, Illinois.

## INTRODUCTION

The impact vulnerability of explosive weapon systems has been studied during recent years by field tests in which the aim has been to bracket the "critical" impact velocity, hereby defined as the minimum weapon impact velocity which causes a violent explosion. 1,2,3,4 Laboratory experiments have formed the basis for a method of predicting the critical impact velocity; the method has been successful with weapons of significantly different designs.

The sensitivity test used for these investigations consisted of squeezing relatively large unconfined cylindrical high explosive samples (1/2 lb-20 lb in weight) between a rapidly moving plate and a massive stationary anvil. Large samples were utilized since the considerable amount of data on the impact sensitivity of explosive charges of the order of grams in weight could not be accurately extrapolated to determine the response of large charges to impact. Thus an experiment was designed with the intent of simulating in an idealized laboratory setup the conditions imposed upon the HE in an impacted weapon (the explosive confined between two materials of high acoustic impedance). Further the nature of the loading in the laboratory experiments is such that the explosive is initiated after a relatively slow crushing process, as occurs in situations of severe accidental impact such as transportation accidents or jettison from aircraft, rather than being promptly initiated by a shock process. Initiation of explosion occurs at much lower stress levels by the crushing process than by the shock process.

The geometry of the experimental system was made simple so that data obtained regarding the physical quantities involved in characterizing the impact phenomena could be employed to predict the impact response of various weapon configurations. (Fig. 1)

Scaling relationships for 9404 PBX and H-6 have been established. Tritonal is presently being evaluated. These relationships showed that the minimum impact velocity required to initiate an explosive reaction is strongly dependent upon the dimensions of the test specimen and that this "critical" minimum impact velocity increases with increase in size approaching a limiting velocity of about 500 ft/sec for 9404 PBX and 600 ft/sec for H-6. Thus under conditions of "gentle" impact a single number does not characterize the impact sensitivity of a large diameter sample of an explosive material. Rather, there is a relationship between the thickness of charge and critical impact velocity, as shown in Fig. 2. In region A of Fig. 2, we see that above a certain velocity (~500 fps, for 9404 PBX), the explosive always detonates high-order. For a much lower impact

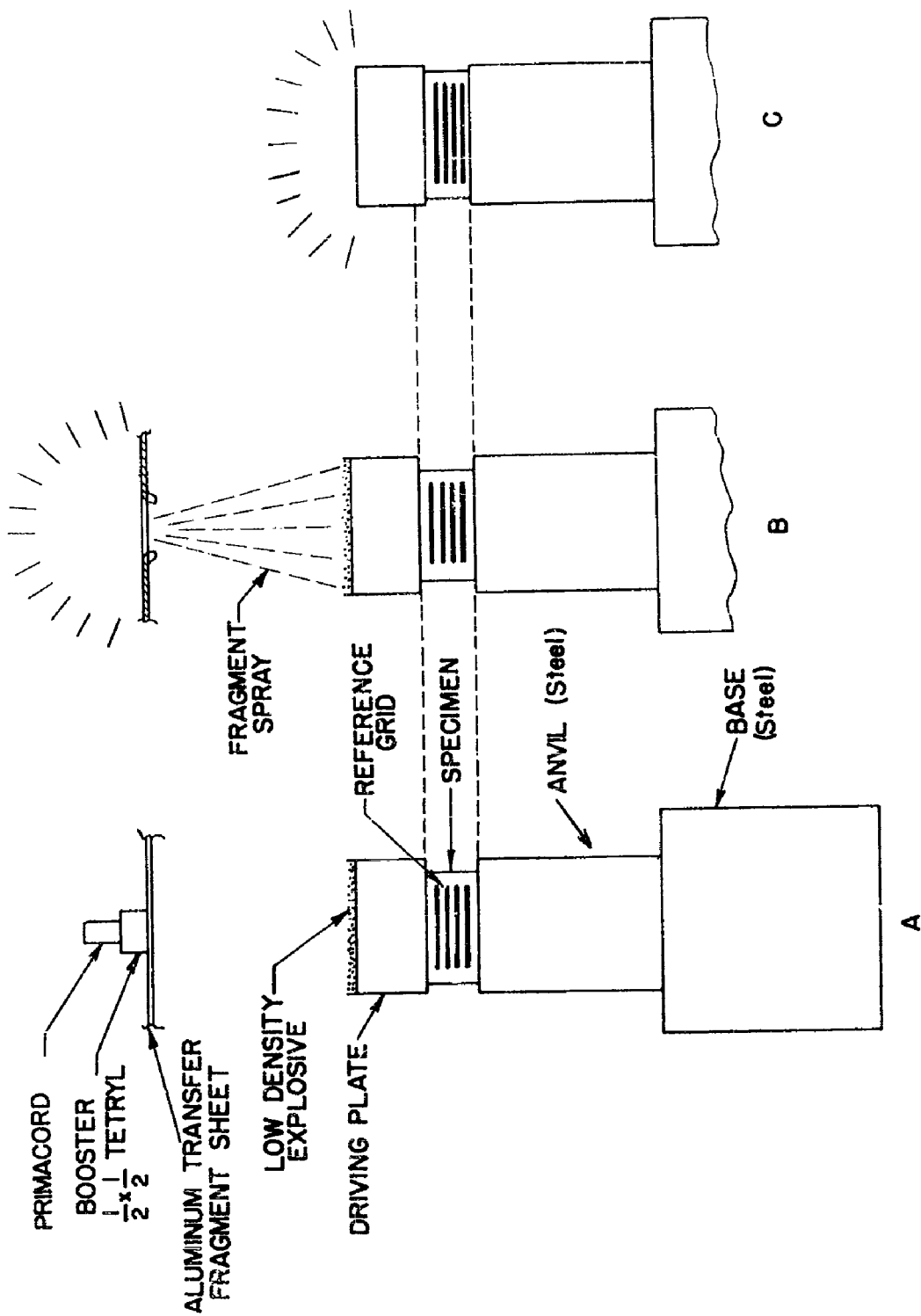


Figure 1 Test Arrangement

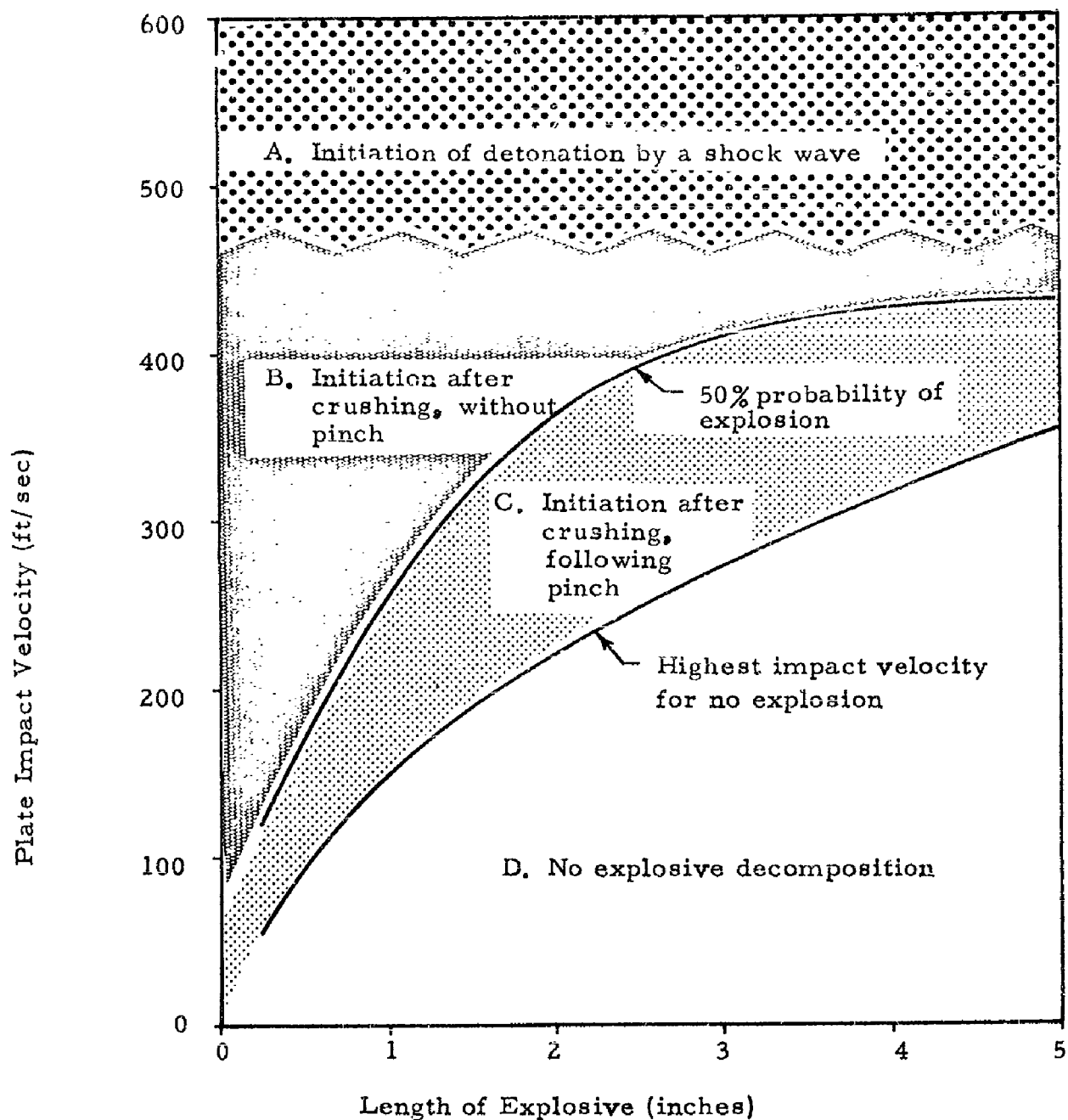


Figure 2 Critical Impact Velocity as a Function of Explosive Length, for 6-inch Diameter 9404 PBX Billets Impacted by 6-inch Diameter x 1-inch Thick Steel Plates

velocity, region D, which depends upon explosive charge size, no chemical reaction is obtained. For intermediate impact velocities, regions B and C, the explosive reaction is generally less violent than the high-order detonations of region A. Further this graph illustrates the experimental results that, for a given diameter, as the thickness of explosive billet increases an increase in impact velocity is required to cause an explosion. A limiting impact velocity, (~500 fps, for 9404 PBX), is reached which corresponds approximately to the velocity of impact required for the shock initiation of a detonation.

High speed framing camera photographs of impact and subsequent initiation of large billets of explosives yielded information on some of the processes involved during impact. In region A of Fig. 2, high-order detonations occur at impact velocities above 500 fps and calculated pressures of the order of 10 kilobars, the explosion occurs within 10 microseconds after impact and during the first passage of the nonreactive stress wave through the explosive billet. In some configurations detonation occurs immediately upon reflection of this stress pulse from the back-up anvil. (Fig. 3,4)

In region B initiation of explosion occurs at impact velocities less than 500 fps and after some small amount of crushing of the explosive billet. The explosion which may be a violent deflagration or detonation usually occurs within 100 microseconds to one millisecond after impact. (Fig. 5)

In region C, which we consider to be the initiation threshold region, explosions occur only after the explosive is crushed to a very thin layer (pinch condition). This has been observed to occur within one to three milliseconds after impact for the plastic bonded HMX-based materials (Fig. 6).

Explosions do not occur in region D, the no-go region. (Fig. 7)

Finally, we observe explosions occurring external to the original charge diameters and after complete crushup of the billet. Here the explosion originates in the explosive dust which was extruded from between the colliding plates. This type of reaction can cause considerable damage and noise. (Fig. 8)

A single mechanism may be responsible for initiation during crushing impact, if the charge thickness affects the operation of this mechanism in such a way that a greater velocity is required for the initiation of thick charges than for the initiation of thin charges. Alternatively, several mechanisms may be important. The prevailing mechanism may change as a function of impact velocity

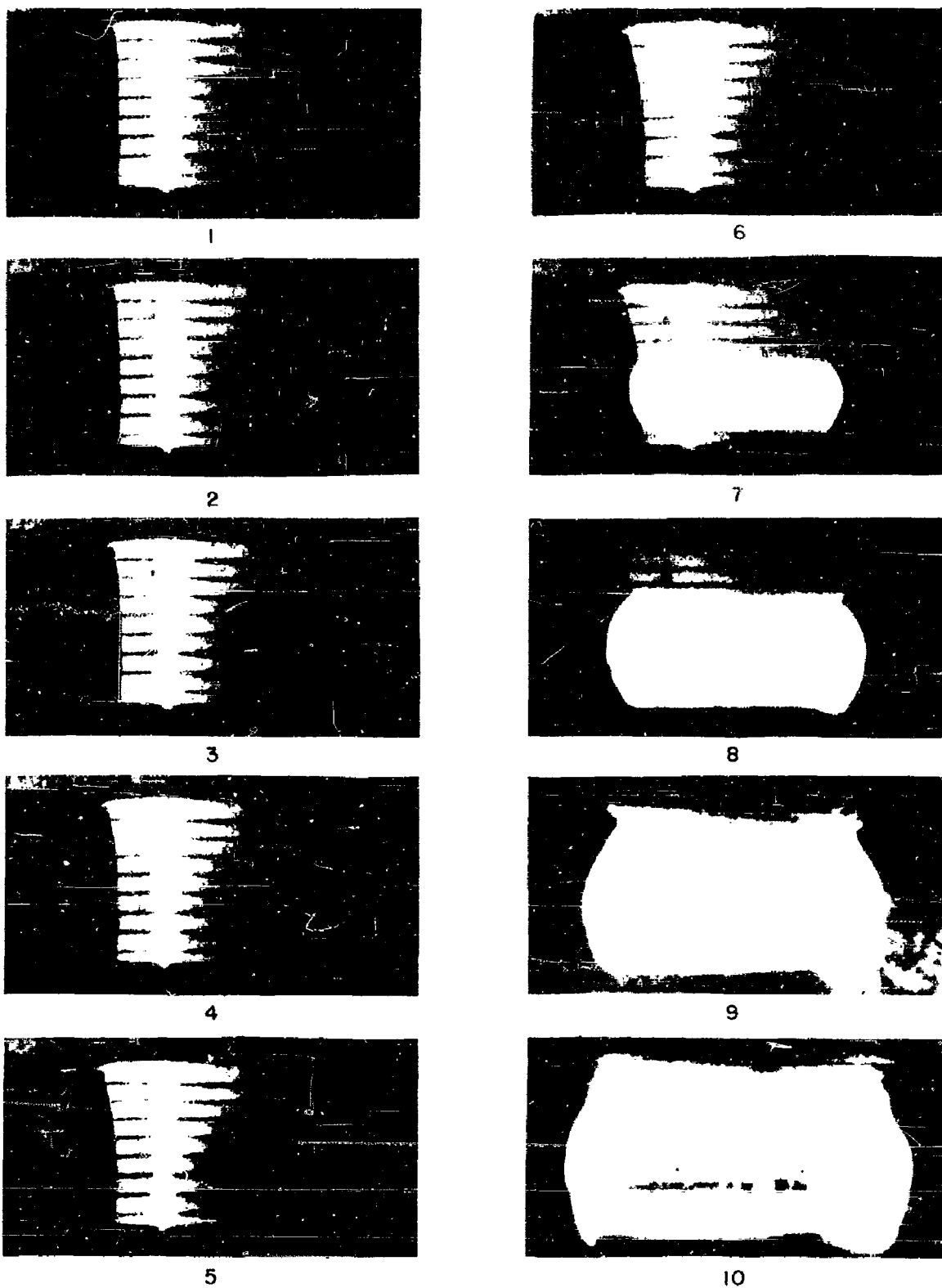


Figure 3 Framing Camera Sequence Showing Forward and Reverse Propagation of a Detonation in 9404 PBX, 1 Microsecond between Frames

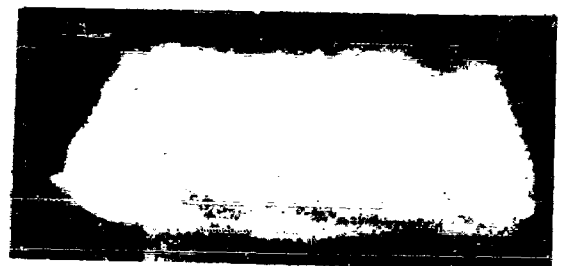
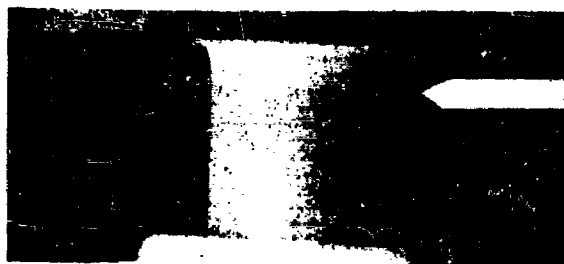
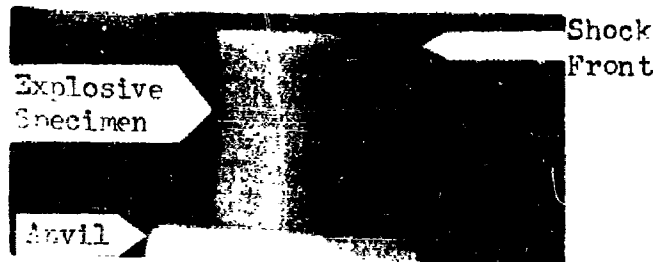


Figure 4 Framing Camera Sequence Showing Impact Initiation of an Unconfined Comp B Cylinder 3-inches Diameter x 3-inches High, 4 Microseconds between Frames

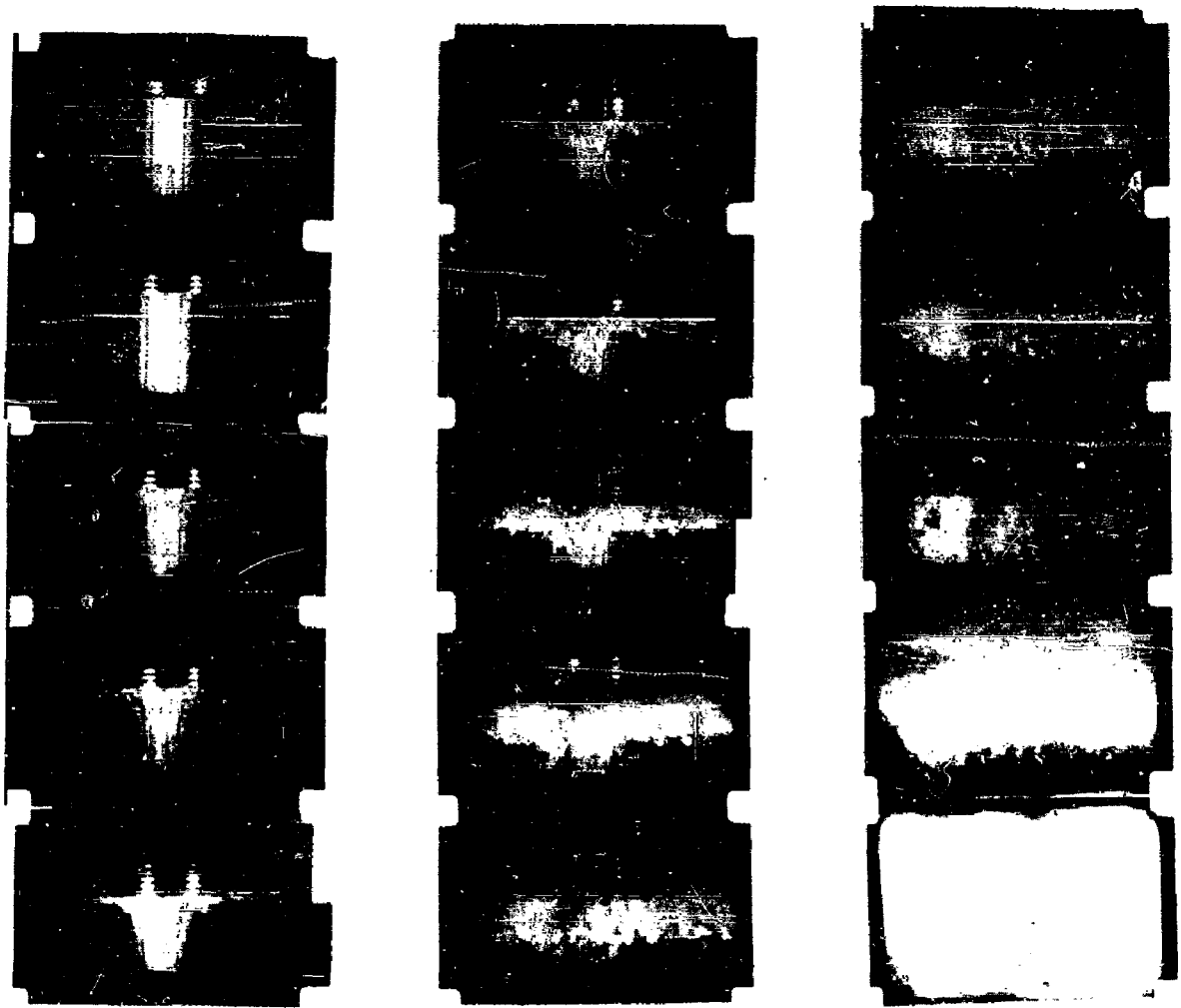


Figure 5 Framing Camera Sequence Showing Impact Initiation of an Unconfined HMX-Base Plastic Bonded Explosive 2-inches Diameter x 4-inches High, 150 Microseconds between Frames, Impact Velocity 140 Feet Per Second



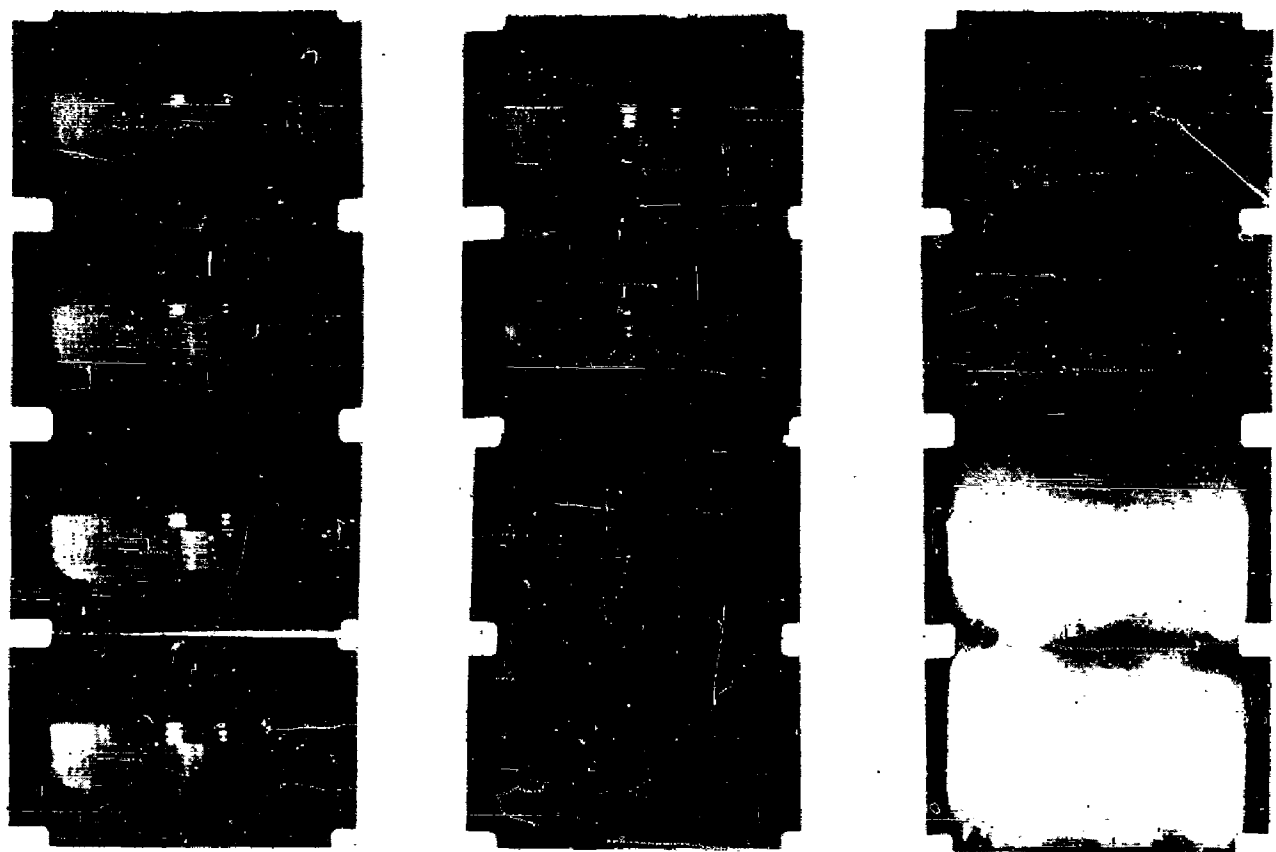


Figure 6 Framing Camera Sequence Showing Impact Initiation of an Unconfined HMX-Base Plastic Bonded Explosive. 2-inches Diameter x 1.75-inches High, 150 Microseconds between Frames, Impact Velocity 135 Feet Per Second

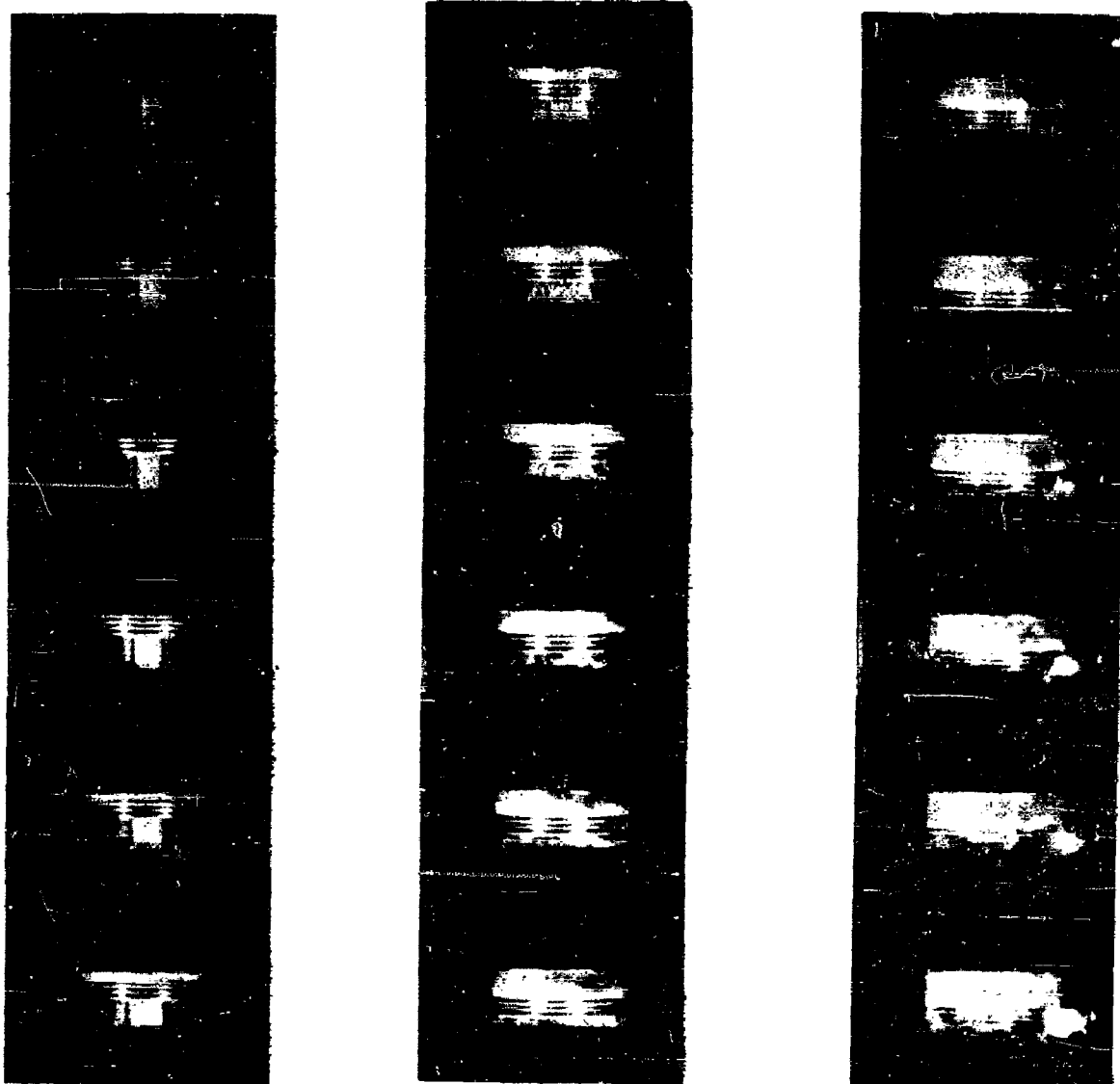


Figure 7 Framing Camera Sequence Showing Complete Crushing, Without Explosion, of an HMX-Base Plastic Bonded Explosive 2-inches Diameter x 1.75-inches High, 150 Microseconds between Frames, Impact Velocity 75 Feet Per Second

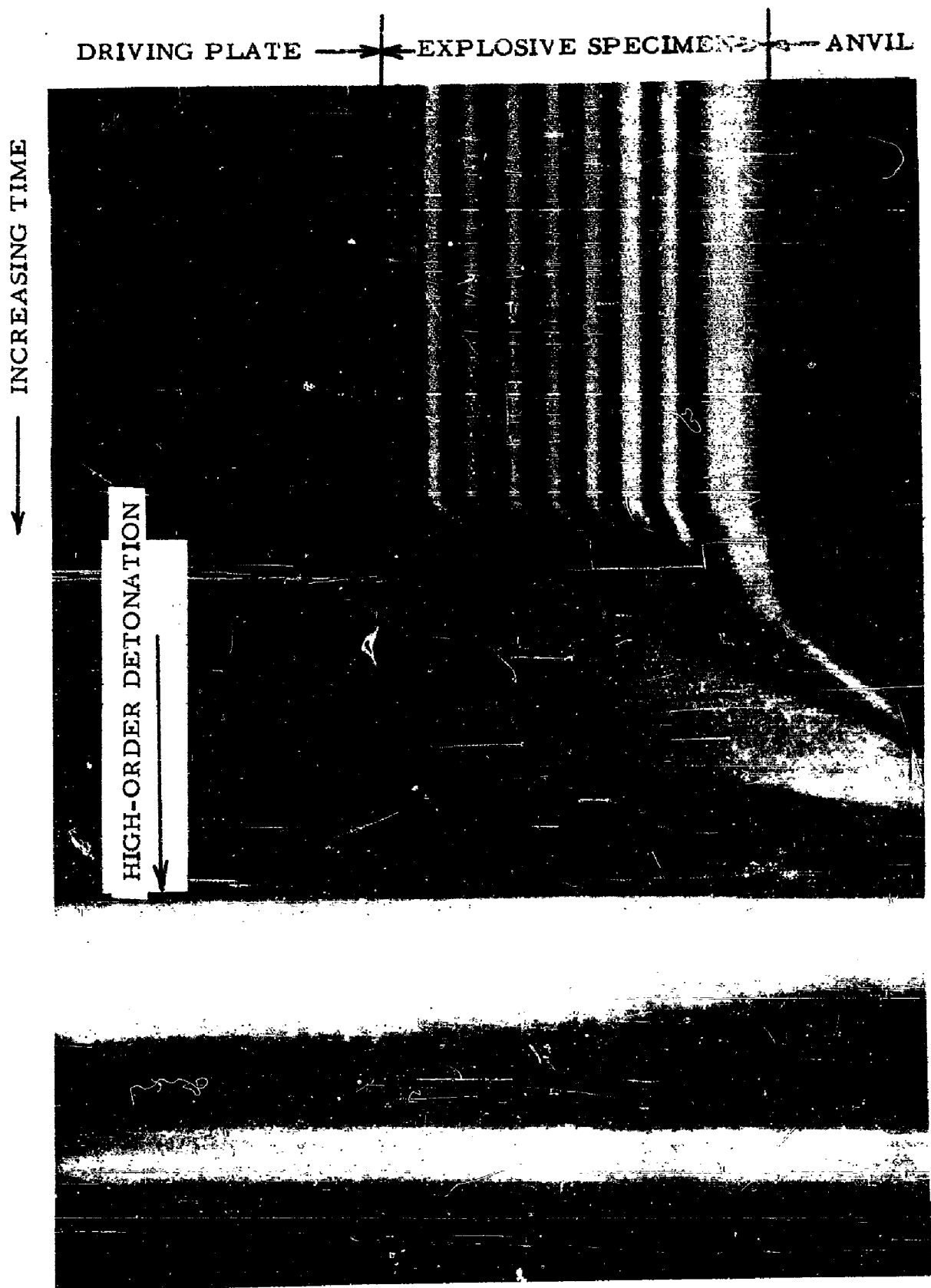


Figure 8 Streak Camera Record of a Dust Explosion Following Complete Crushing of Composition B, 3-inches Diameter x 1-inch High, Impact Velocity 600 Feet Per Second

at a given explosive specimen thickness, or may change as a function of thickness along the initiation (50 percent probability of reaction) curve. One cannot deduce simply from the shape of this curve or from high speed photographs of the crushing process and subsequent initiation whether a single initiation mechanism operates over the entire range of impact conditions, or whether different mechanisms are involved for diverse loading conditions. The particular mechanism that operates in a given application depends upon the explosive configuration, its environment and its composition (as reflected in its non-reactive material properties such as brittleness, thermal diffusivity, explosive crystal form, etc.). Therefore the relationship between impact velocity and charge size which characterizes the sensitivity to impact of one material would not be expected to be the same for another explosive composition. One should expect that the curve representing this relationship for uniform impact loading of one explosive material would be shifted or changed in shape for an explosive of a different composition or for the same explosive subjected to a concentrated load.

Figure 2, which we consider to be the scaling laws which govern the impact sensitivity of a particular explosive (in this case 9404 PBX) has been used with an analytical procedure which accounts in part for the effects of weapon design. This has made it possible to predict the impact velocity required to cause initiation of the high explosive component of special weapons. In tests in which complete weapons were dropped from flying aircraft or were impacted against a concrete target through the use of a rocket sled, the prediction calculations yielded values which were within 20% of the bracketed critical impact velocity in case of six out of seven weapon types tested. In the instance in which the prediction was much higher than the observed critical impact velocity, it was observed that a concentrated rather than distributed loading condition existed within the weapon. The stress concentration was apparently responsible for the increased sensitivity of the weapon.

It should be noted that the weapon tests showed that reactions originated in thin layers of explosives within the weapons, which bears out the conclusion from Fig. 2 that thin layers are much more sensitive than thick layers of explosive.

#### MODEL FOR PREDICTING IMPACT INITIATION OF THE EXPLOSIVE COMPONENT IN A WEAPON

The following is a description of the model used to predict the critical impact velocity of a weapon.

Upon impact of a weapon against a target, the sudden application of load causes deformations and stresses which are not immediately transmitted to all parts of the body. Remote portions of the weapon may remain undisturbed for some time. From the point of application of load to the location of the explosive component within a weapon, the propagation of the disturbance (stress wave) caused by the impact crosses several interfaces.

Because stress wave propagation is affected by the physical properties of the medium through which the wave is traveling, it will be modified upon crossing interfaces between different materials. At each boundary between different materials in the weapon the amplitude of the initial stress as well as the particle velocity is modified. Consequently, the shape and amplitude of the pulse may be expected to be altered by the time it reaches the explosive.

Since the impact sensitivity of the explosive may be characterized by the velocity of impact and it is possible to calculate the amplitude of the stress wave during propagation through the many inert materials in the weapon, predictions are in fact possible. The experimentally determined velocity required for the initiation of explosive specimens in laboratory tests provides the empirical input needed to compute the weapon impact velocity which would cause an explosive reaction.

In summary, the explosive response is determined experimentally under environmental conditions designed to be as simple as possible. These experimental results may then, by proper computations, be applied to weapon configurations. The prediction calculations are based on consideration of the geometry of the inert and explosive materials which make up the weapon represented as a one-dimensional layered system. Stress wave transmission through the composite is calculated for a given initial impact velocity; stresses and velocities within the explosive components are computed. The material velocity is assumed to cause a weapon reaction similar to the reactions of laboratory test specimens at an equal explosive material velocity.

Basically the prediction model has been effective because it makes use of results of laboratory impact experiments on large explosive charges whose configurations were similar to those found in weapons. Further the nature of the loading in the laboratory experiments was such that the explosive was initiated after a relatively slow crushing process distributed over a rather large area, as occurs in the severe impact of weapons, rather than being promptly initiated by a shock process. Initiation of explosion occurs at much lower stress levels by the crushing process than by the shock process.

The impact sensitivity data are then used in conjunction with an analysis of the propagation of stress waves through the weapon. The extent to which the impact load is modified by the time the disturbance arrives at the weapon's explosive components is calculated. Knowing the intensity of the modified pulse for a given impact velocity, the scaling relationship (Fig. 2) determined by the laboratory experiments indicates whether or not initiation of the explosive will occur.

The limitations of the prediction model lie in the degree or extent to which the several assumptions which are made are valid. The following is a brief discussion of the assumptions which require further examination.

### Material Properties and Wave Interactions

In the analysis only the first passage of the elastic stress wave through the materials is considered. Further, elastic material properties are assigned to the foams, rubber and plastic materials. However, under impact conditions which result in explosion, most of the materials in the weapon are stressed beyond their elastic limit. The effects of the propagation of plastic waves and unloading waves as well as wave interactions contribute to the stress-particle velocity distribution in the explosive.

When an impact load is applied to the weapon, which is assumed to be a one-dimensional layered system, discontinuities of stress and strain will propagate through the weapon, from the point of impact. When these disturbances reach a different material in the layered system, they are partially transmitted to the forward layer and partially reflected back into the previously stressed layer. This process of multiple reverberation and interaction continues until the mechanical state predicted by elementary mechanics is approached in a gradual and in general, discontinuous fashion.

Materials loaded at stresses below their dynamic yield point, in their crushing or strain-hardening region, or in their compacting region, will develop acoustic waves, dispersive compression waves, and plastic or shock waves respectively. After a material has been compressed to a particular state, unloading can take place either through a single discontinuity or through a series of unloading wavelets, depending upon the equation of state of the material in unloading. Obviously, accurate information concerning the dynamic stress-strain curves, the equation of state, must be used in order to specify completely the transient phase of motion in a test material.

Impingement of a layered weapon upon a target, composed of materials with complicated equations of state, can induce a multiple wave system which is further complicated in internal reflections and external boundary reflections. For certain combinations of stress levels and wave forms, the stress wave fronts can become still more numerous because residual strain discontinuities are generated by stress wave interactions, and provide additional interfaces for subsequent wave reflection.

In order to construct the overall solution to this type of problem, it is only necessary to consider a single interaction at a time, to deduce the stress and strain states resulting from the particular interaction and then to seek the earliest subsequent interaction and perform the same type of calculation.

The solution of each wave interaction problem appears complex in its final form; however, the construction of the network is readily obtained by considering the individual interactions. Each interaction, no matter what combination of stress or strain waves interact, is handled in the same way. However, a general procedure can be followed which takes into account all possible complexities of any type of interaction. This general procedure has recently been programmed for machine computation, but has not as yet been applied to the weapon impact problem.

#### Impact Sensitivity of Explosives

The scaling relationship shown in Fig. 2 characterizes the sensitivity to uniform impact loading, of one material 9404 PBX, as a function of charge thickness. Such a curve will be shifted or changed in shape for an explosive of a different composition, or for the same explosive subjected to a concentrated load. Curves of this type may be applied to predictions of weapon vulnerability without an understanding of the details of the mechanisms responsible for initiation, provided that for a given explosive thickness the same mechanism controls the explosive's response in both the laboratory and weapon tests.

#### Structural Strength

In the primary or "most vulnerable" test condition, the structural strength of the weapon system was assumed to be negligible. This assumption was found to be in reasonable agreement with the weapon test results. However, for those impact attitudes believed to be significantly less vulnerable, by virtue of the apparently greater structural strength of the weapon system, the structure must be accounted for in the prediction analysis.

## ACKNOWLEDGEMENT

Investigations of plastic bonded materials and full-scale weapon tests were supported by FC/DASA under Contracts AF 29(601)-2133, DA-29-044-XZ-885, and AF 29(601)-1959. Work on TNT-based materials is being supported by Eglin Air Force Base, Detachment 4, Research and Technology Division, Air Force Systems Command under Contract AF 08(635)-3789.

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Dr. Price: Do you attribute the greater sensitivity of the thin layers to the multi reflection of the stress between your impacting plate and the anvil and the higher stress levels at which it is reflected?

Mrs. Napadensky: For those points in the neighborhood of the 50% curve of Figure 2, the increased sensitivity is only in part due to the influence of the multiple reflections of the stress wave. Initiation of an explosion occurs only after the explosive has undergone a considerable amount of crushing. The time from initial impact to explosion is of the order of 1 millisecond. This is sufficient time for hundreds of reverberations of the stress wave between the plate and anvil. During the first 100 microseconds, or so, the explosive has reached an equilibrium pressure, so we do not believe that the stress waves are the primary stimulus. The problem of isolating the forces which dominate or control the process of initiation after crushing is a difficult one.

Dr. Price: Have you looked at any other less sensitive material to crushing besides tritonal?

Mrs. Napadensky: No, but we have just begun an IITRI-funded investigation of the sensitivity of propellants to crushing impact. We have not yet fired our first shot.

Mr. Hart: Clarification on one statement that you made. You mentioned concentrated loads, could we apply this to something such as a point target or spike target vs a flat plate or level target? Does DASA or does your laboratory plan to extend your study to point concentrated targets?

Mrs. Napadensky: The data presented here and all experiments we have carried out thus far have utilized a flat plate and back-up anvil and hence can be applied only to relatively level or flat targets. We hope to begin, within the next few months, a study of the impact sensitivity of certain explosives to concentrated loads. These tests will be directed toward simulating either some likely target or some geometric configuration within a weapon, such as a bolt, which may crush into the explosive after impact.

Mr. Hart: You stated that your graph for 9404 PBX does not apply to concentrated loads. Does it follow the same sort of relationship? Do you have any idea?

Mrs. Napadensky: I'd expect that the explosive or propellant would appear to be more sensitive to a concentrated load. However, I don't believe that the results for concentrated loads would be in any way similar to the curve for uniform loads, relating thickness of explosive to critical impact velocity.

Mr. King: You mentioned that for the higher impact velocity that the initiation was by shock. In the lower velocity regions do you attempt to correlate the ignition phenomena with any of the hot spot theories or adiabatic compression of gases or things of that sort?

Mrs. Napadensky: We have made calculations that indicate that the hot spot theory may apply and that the calculated temperature rise due to adiabatic compression of trapped gas bubble is sufficient to cause initiation provided certain other assumptions are met, however, we have by no means proved that it does apply. This question is discussed in a paper by Napadensky, Stresau and Savitt, "The Behavior of Explosives at Impulsively Induced High Rates of Strain," presented at the Third Detonation Symposium, ONR Symposium Report ACR-52, Vol. 2, 1960.

Our more current opinion is that flow phenomena are involved. Explosive material driven laterally outward from the unconfined billet during impact crushing has been observed to have very high lateral velocities. A material balance suggests that velocity gradients through the extruding explosive must be especially severe whenever the billet has been crushed to a small thickness (the pinch condition). Substantial local temperature rises may occur as a result of viscous heating within the solid explosive. The crushed remains of color-coded explosive samples which did not react (Ref. 2) showed significant mixing of explosive elements originally located in different regions of the sample; this suggests the possibility of turbulent solid flow, as suggested by Dr. Price.

## PNEUMATIC CONVEYING AND REMOTE PACKAGING OF BASE-GRAIN CASTING POWDER

John T. Owings, NPP Indian Head, Md.

The process for manufacturing base-grain casting powder is similar to the solvent extrusion process by which gun propellants are manufactured. The various ingredients (nitrocellulose, nitroglycerin, aluminum, ammonium perchlorate, and a stabilizer) are mixed in a horizontal sigma-blade or vertical mixer. The resultant mass is extruded as solid strands which are subsequently cut into solvent-wet propellant grains (base-grain casting powder) of the desired length. Figure 1 is a flow diagram on the manufacture of high-energy casting powder.

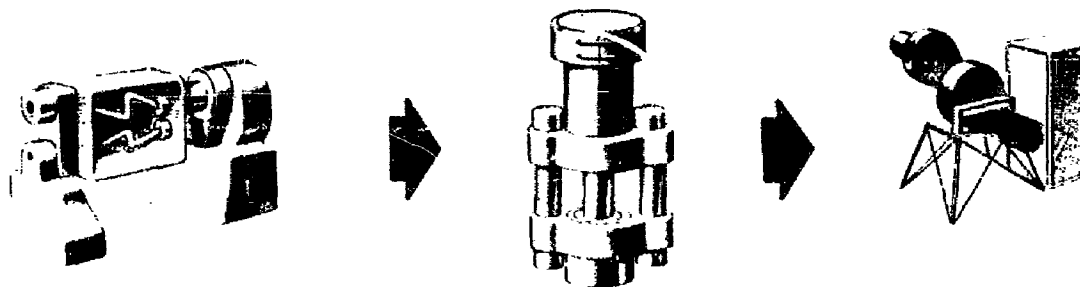


FIGURE 1. FLOW DIAGRAM FOR MANUFACTURE OF CASTING POWDER

The solvent-wet casting powder is processed through the finishing area to reduce the solvent content to the desired level before further processing through glazing, screening, blending, and packing. Finishing-area operations require repeated handling of the finished casting powder and this involves the exposure of personnel to large quantities of explosives.

The purpose of this paper is to review the studies conducted and equipment installed at the Naval Propellant Plant to permit remote processing and handling of high-energy solid propellants. Personnel safety was the prime factor considered in the design and installation of remote handling facilities. However, it should be noted that every change made to improve personnel safety resulted in improved quality and reduced cost.

In the transition from low-energy to high-energy solid propellants, the major area for safety improvements in the finishing area was considered to be the elimination of manual handling of "in-process" propellants in regular shipping containers. These improvements were made by incorporating improved

material handling equipment to permit automatic handling and remote control where possible. The equipment included (1) portable drying racks and trays to replace bin-type drying, (2) portable grain containers equipped for remote discharge, (3) pneumatic conveying systems for conveying between operations, and (4) automatic weighing and conveying equipment.

The drying rack is a portable drying bin constructed of magnesium to hold twenty aluminum trays. The drying rack replaces manual handling and cross stacking of trays and the fixed bin-type dryers. The drying rack is designed for uniform distribution and optimum contact of the drying medium (heated air) with the casting powder. The rack is equipped with conductive rubber wheels for protection against accumulation of static electricity and to eliminate friction and pinching of propellant between the wheel and floor. Most propellants are nonconductors and require special safety precautions to prevent the accumulation of static electricity. The drying tray is a die-formed perforated aluminum tray with a capacity for approximately 20 pounds of casting powder. A uniform layer of casting powder is weighed in each tray at cutting, placed in a portable drying rack, and transported to the drying operation.

After drying, the casting powder is conveyed between operations in a portable grain container. The grain container (Figure 2) has a capacity of approximately 1000 pounds and is constructed of aluminum and in such a manner that a bed depth of 30 inches of propellant is never exceeded. The container is a multipurpose container equipped with valves to permit remote discharge either at a rapid rate or at a slow controlled rate.

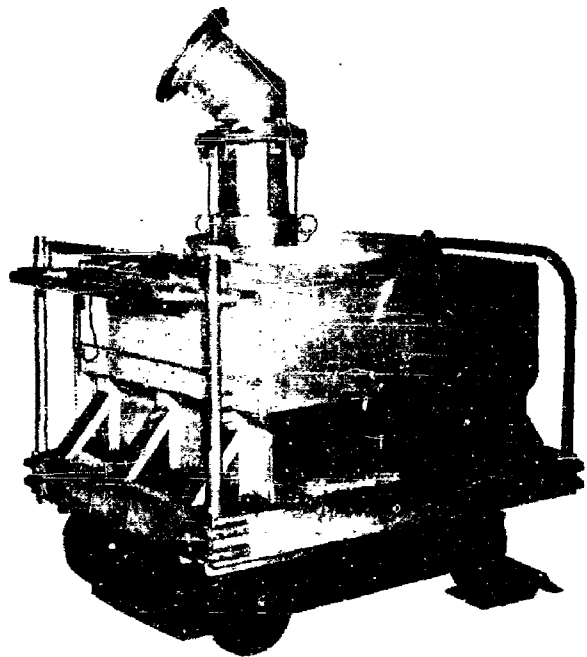


FIGURE 2. PORTABLE  
GRAIN CONTAINER

### PNEUMATIC CONVEYING SYSTEM

The most important improvements were made by incorporating pneumatic conveying systems in the finishing area that eliminated the manual handling and transporting of casting powder between glazing and screening and between blending and final packing.

The design of the pneumatic conveying systems installed at the Naval Propellant Plant took into consideration the results and recommendations of investigations conducted throughout the propellant industry relative to pneumatic conveying of finished propellant. Some of the key recommendations include minimum conveying velocity, intermittent or interrupted flow, grounding and humidity control to aid in dissipation of static electricity, valving to control feed to the conveying system, and piping arrangements to prevent damage of propellant grains due to impact.

These recommendations are based on results of experimental investigations conducted by Ravenna Arsenal, Alabama Ordnance Works, Allegany Ballistics Laboratory, Indiana Arsenal, Radford Arsenal, the Naval Propellant Plant, and U. S. Hoffman Machinery Corporation.

The pneumatic conveying systems (Figure 3) are vacuum or suction systems designed to convey casting powder at a rate of 300 lb/min and at a velocity of 7500 ft/min. The conveying system consists of a centrifugal exhaustor, peristaltic feeder valves, high efficiency cyclone separators, and a wet and dry dust collection system.

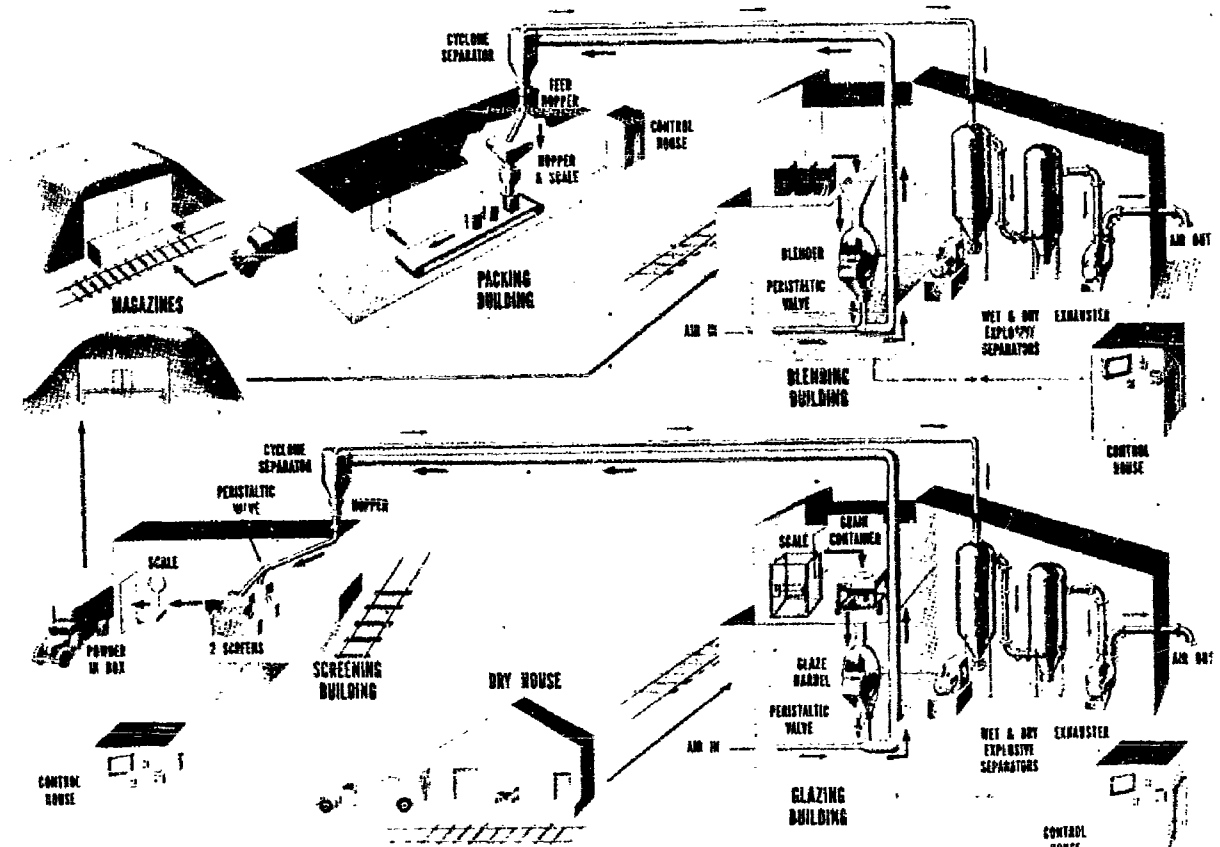


FIGURE 3. FLOW DIAGRAM OF PNEUMATIC CONVEYING SYSTEMS BETWEEN GLAZING, SCREENING, BLENDING, AND PACKING OPERATIONS

The exhauster is a multistage centrifugal-type exhauster (U. S. Hoffman model 38506) powered by a 75 hp motor and operates with a suction of approximately 8 inches mercury. The exhauster is equipped with three radial aluminum impellers and is operated at 3500 rpm.

The safety requirements for valving the flow of high-energy solid propellants were necessarily very rigid. The valve is required to operate accurately through several cycles per minute to permit interrupted flow of propellant and at the same time be free of friction and pinch points. The valve selected was an 8-inch peristaltic valve with pure gum rubber diaphragms.

The valve is controlled by alternately applying vacuum and air to the diaphragms to open and close. Timers are used to permit the peristaltic valve (Figure 4) to be open from 1 to 6 seconds. In actual practice, we have controlled propellant flow at 100 lb/min by setting the valve to be opened 1.5 seconds and closed 4.5 seconds. This resulted in an interrupted powder flow of 10 pounds each 6 seconds with each increment being separated by a distance of approximately 75 feet. Interrupted flow was incorporated to prevent upstream propagation in the event of ignition.

The valving arrangement is designed with safety interlocks to fail-safe in the event of loss of electricity or loss of compressed air. It is important to note that the Naval Propellant Plant has processed greater than 25 million pounds of high-energy solid propellant through pneumatic conveying systems without incident.

Another prime concern in the safety design of the pneumatic conveying system was the high efficiency cyclone separator for separating the casting powder from the air stream. The cyclone separator (Figure 4) was designed to

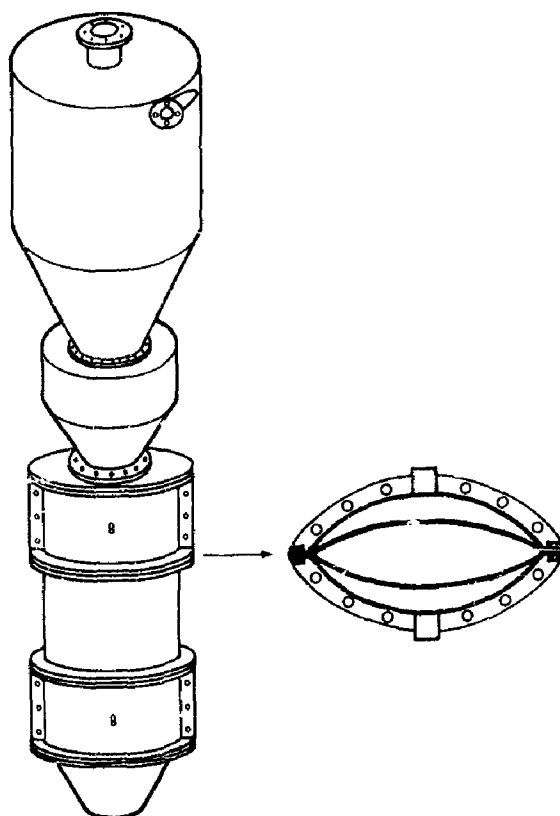


FIGURE 4. CYCLONE SEPARATOR  
AND PERISTALTIC VALVE  
ARRANGEMENT

operate at an efficiency of 99.5% to prevent casting powders containing finely divided metals from carrying over into the wet dust collection system and exhauster. In actual practice the high efficiency cyclone separator has been 99.996% efficient.

The casting powder flows by gravity from the glazing and blending barrels through a single peristaltic valve into an 8-inch stainless-steel line and the air stream. The powder is separated from the air stream through the cyclone separator and peristaltic valve arrangement and discharges by gravity into two screening machines. The powder is screened and weighed into shipping containers on a continuous and automatic basis.

The air with powder fines and dust is returned through a wet and dry dust collection system for cleaning prior to exhausting. The wet separator (Figure 5) is designed specifically for explosives to cause the air and in-trained dust to mix thoroughly with water, thus trapping all collected material as wet sludge. The wet separator is designed for self-cleaning. The internal baffles are arranged to cause the air to break up into fine bubbles and change direction three times while submerged.

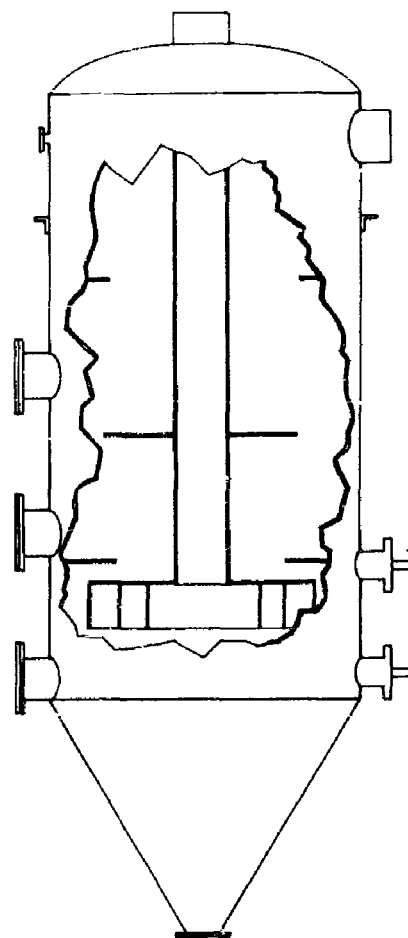


FIGURE 5.  
WET PRIMARY  
EXPLOSIVE SEPARATOR

The secondary separator (Figure 6) is a combined centrifugal and inverted filter bag-type collection system. It is equipped with 60 napped cotton moleskin bags. The bags are rigidly mounted with no provisions for mechanical vibration. In practice, they require a minimum amount of cleaning. As an example, the total amount of dust collected after conveying 100,000 pounds of explosives is less than 100 grams.

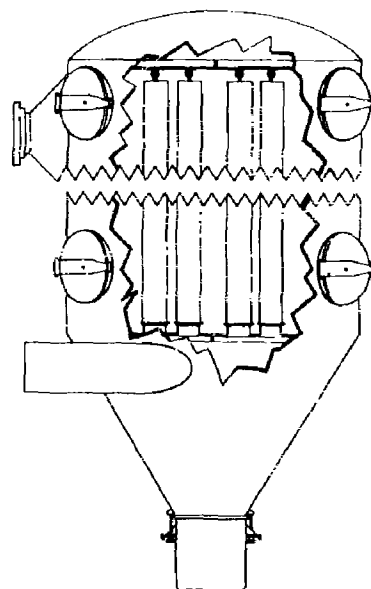


FIGURE 6.  
SECONDARY SEPARATOR

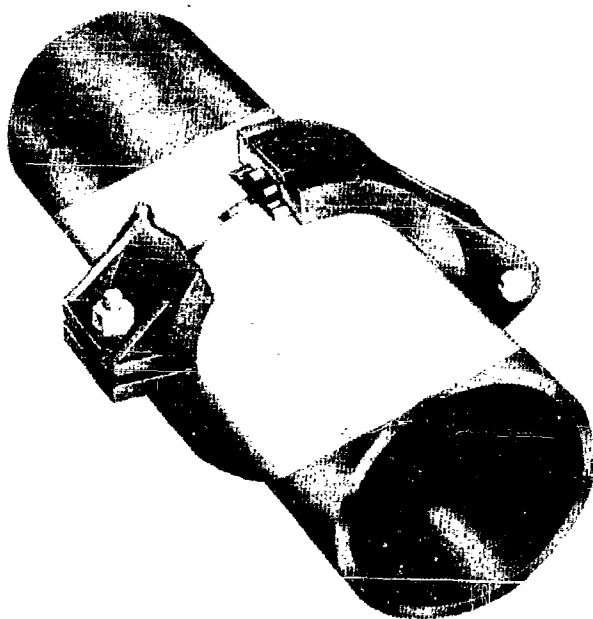


FIGURE 7. SWEPCO  
SELF-ALIGNING COUPLING

The explosives conveying line in each system is an 8-inch-diameter pipe, approximately 400 feet in length and is constructed of 304 stainless steel. The return lines are 10-inch-diameter black iron pipe. The pipe line bends are held to a minimum and are long radius bends.

The couplings (Figure 7) used in the explosive conveying line are required to have a smooth surface internal to the pipe and to be free of pinch points. The coupling used is a Swepeco self-aligning coupling.

The motors and electrical controls are Underwriters approved explosion-proof (Class I Group D and Class II Groups E, F, and G).



## AUTOMATIC WEIGHING SYSTEM

The automatic weighing systems are interconnected with the pneumatic conveying systems. The weighing systems consist of powered roller conveyors for conveying empty containers to and full containers away from the weighing station. The weighing system was fabricated and installed by the Toledo Scale Company. The weighing system is adjustable for weighing 1 to 225 pounds with an accuracy of  $\pm 4$  ounces. The system weighs one container at 125 pounds each 28 seconds.

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Mr. King: I presume you heated and cleaned and dried the air in the conveying system. Do you have any idea what the relative humidity was after you finished this? I don't think you can get off the hook by saying its damp in Washington.

Mr. Owings: I said we have made no attempt to control the humidity. The relative humidity in Washington is 60% most of the year, around or better.

## PNEUMATIC MIX PROCESS

Jay Carsey

U. S. Naval Propellant Plant, Indian Head, Md.

The U. S. Naval Propellant Plant (NPP) has as a primary mission the constant search for better, more efficient, less costly, and safer solid propellant processes. This afternoon I would like to describe a process which was invented and is in development at NPP—the Pneumatic Mix Process. We feel it represents an advance in the state-of-the-art of propellant processing.

Before I go into the process itself let us briefly survey the evolution of the solid processing industry. One of the unique features of NPP is that it contains, within its physical plant, a cross-section of this evolution. The solid processing industry emerged from the old gun propellant manufacture in which nitrocellulose and a few additives are extruded into a form designed to achieve a proper burning rate. From this was the natural emergence of the extruded solid propellant in which larger forms are extruded and more complex ingredients used. Today's Asroc, Zuni, and Rapec rocket motors are still manufactured with this earliest process. However, the extruded process soon was limited. The heat and pressures involved in processing excluded the incorporation of the more energetic ingredients as they were discovered. The industry then developed the cast double-base system in which small granules of casting powder were extruded and these immersed in an energetic solvent and cured at elevated temperatures. This process, refined over a 10-year period, achieved a sophistication that saw it ultimately accepted as producing a product safe enough for the confines of a nuclear submarine. With this process, specific impulses in the 250's were achieved, but at about that point a limit was reached. As attempts were made to pack more energy in the same volume, safety hazards in the process became prohibitive, particularly in the mixing portion of the process. In addition, the process was complex and expensive. The industry found itself swinging toward the composite system in which energetic solids were incorporated in a polymeric binder and the motor cured in practically a one-step operation.

But all of these processes were essentially batch processes; and the solid propellant industry, anxious to reduce cost, simplify control, and eliminate personnel hazards, has searched constantly for continuous techniques for processing solid propellant. NPP, a Navy in-house facility, has actively participated in this search.

One important point injected here—one real advantage of the solid propellant system is that once the energetic ingredients have been cured in their matrix, one

may handle it as an intrinsically safe material. Therefore, if a safer means of processing the ingredients to this final stage is discovered, a rather significant step has been taken in reducing the over-all safety limitations of the system.

There have been two general approaches to the search for continuous processing in the propellant industry. One has been the modification of standard equipment, and the other has been the investigation of unique techniques for the processing itself. Pneumatic mix is an example of the latter.

Not to prolong the suspense any longer, I would like to generally describe the process, discuss its safety and engineering advantages, and outline what has been done to date in its development. What we have essentially done in the Pneumatic Mix Process is substitute turbulent gas flow for mechanical mixing. Figure 1 shows, in schematic form, the essence of the process. We have a solids feeder which ejects the solids in a gas stream in the upstream end of a long porous pipe. This porous pipe is designed so that pressure is maintained on the inside of the pipe forcing the ingredients down the center of the pipe away from the walls. A liquid feeder inserts the liquids into the system at the upstream end of the pipe. High velocity gases move the ingredients down the pipe and intimate mixing is achieved by the turbulent gas stream's action on the ingredients. At the downstream end of the porous pipe the mixed ingredients go into a cyclone separator, the gas going off the top and the mixed propellant into some sort of a receptacle.

This is the very briefest description of our process, but it contains some very definite and important safety and engineering advantages. One of the most important safety advantages is that, at any one time in your mixing operation, there is only a very small amount of propellant being actually mixed. One of the major safety disadvantages of mechanical mixing is that there are several hundred pounds of propellant being "worked" by metallic blades and, should propagation occur, a quite formidable bomb is involved. With the Pneumatic Mix Process there is, at any one time in the propellant mix, only a very small amount of ingredients. For instance, in processing approximately 5,000 pounds an hour there is only approximately 1 pound of propellant being mixed at any one time at any one place in the process. This is quite different from having several hundred pounds of propellant being mixed together at one time in one place. This is, therefore, a definite safety advantage of this particular process, and we feel a very important one. A second safety advantage, which is also a very significant one, is that this process has virtually eliminated all forms of mechanical mixing. You have no large blades moving around on the inside of the bowl mixing up the propellant; there are no mechanical moving parts. Another safety advantage is the ability to use complete remote control facilities for the mixing and processing of the particular propellant in process.

# PNEUMATIC MIX PROCESS

## UNIT FOR CONTINUOUS MIXING OF PNEUMATICALLY CONVEYED SOLIDS AND A LIQUID

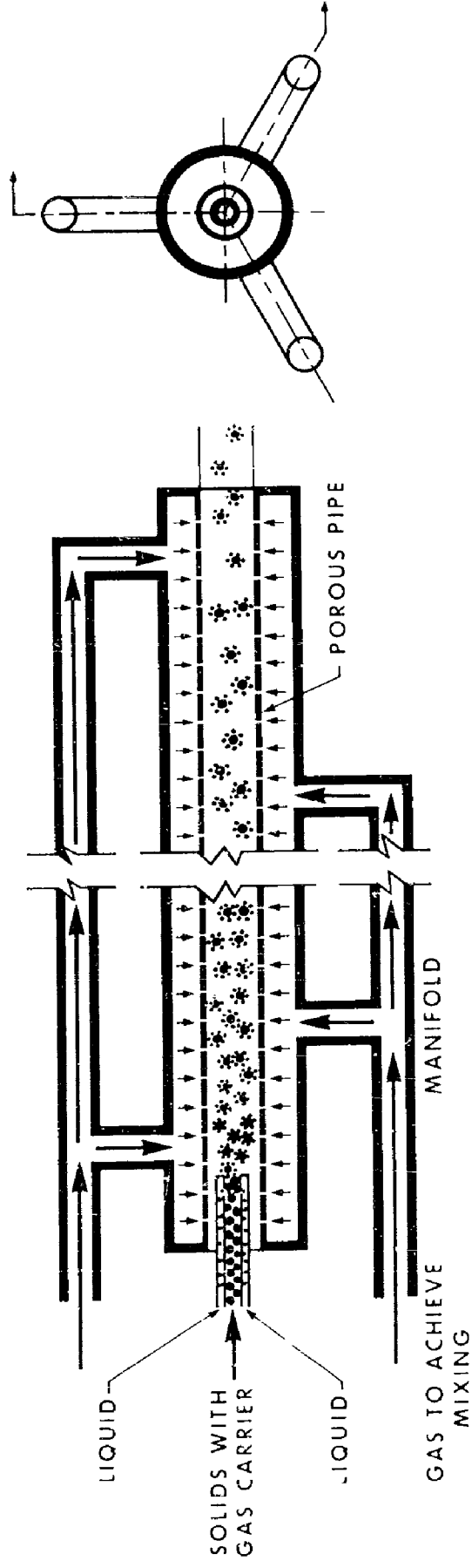


Figure 1

The ability to completely place your operations under remote control is an important step because, even though safety hazards can never be completely eliminated, the industry must continually attempt to eliminate the personnel hazards. By being able to operate completely under remote control, we have gone to the ultimate in personnel hazard reduction. Another potential advantage of this process, from a safety standpoint, is that it seems to have potential for use in processing toxic materials; the air being used throughout the system could be also utilized to flush toxic vapors coming off the mixing process. So there are several safety advantages to this process which we feel are quite significant.

Now I would like to talk about some of the engineering advantages which accrue to this process. One of them is, of course, the cost advantages of such a continuous process. The equipment is relatively inexpensive and easy to maintain. For large amounts of composite propellant, the processing cost per pound would be quite low. The process is also quite flexible; it can be moved from place to place at a small expense. The mixing process could virtually go to the rocket motor, which is the reverse of the standard procedure.

A note at this point about what has actually been done with this process. Feasibility for processing composite propellant has been proven. A pilot plant, with a capacity of approximately 200 pounds per hour, has been made operational, and composite motors containing about 75% solids (ammonium perchlorate) have been successfully processed and static-fired. One of the encouraging discoveries was that the physical properties of the pneumatically-mixed motors were superior to similar ones cast by the conventional method. Also microscopic analysis of cross-sections of the cured grains indicated that a more homogeneous product had been achieved through the pneumatic mix process. This was a pleasant discovery, but no real surprise. It would logically seem that a more intimate, and therefore more homogeneous, mix would be achieved using turbulent gas flow as opposed to mechanical mixing.

Another engineering advantage with composite systems is that the pneumatic-mix process allows the use of ingredients which have shorter "pot lives." The conventional process involves a mixing cycle much longer than that of pneumatic mix and is further lengthened by a transfer step. With the pneumatic mix one can project an extremely quick mixing cycle and thus open the door to the use of the polymeric combinations heretofore prohibited by the nature of the process. Thus, we feel that the engineering advantages of this process are rather imposing. One has a more flexible process which can produce a less expensive, more homogeneous product using ingredients which have shorter "pot lives."

A little more discussion on the safety aspects of the process seems in order here. Before I leave the engineering aspects, however, I do not want to give the impression that all of the engineering problems have been overcome. We have processed a reasonably standard composite propellant on a pilot-plant scale. The engineering challenges are numerous in order to take this to a continuous process on a production-plant scale. However, none that we have foreseen to date would seem to belie solutions.

The safety aspect which has received, and will continue to receive, the most attention is static electricity. We have had some rather comprehensive tests run by our own people as well as outside consultants to gauge what magnitude of electric buildup we are experiencing in our fast gas stream and what needs to be done to minimize the hazard, if indeed a hazard does exist. The speed of the gas itself does a lot to eliminate some of the hazards. The ingredients are moving through the pipe so fast that they figuratively do not have time to be affected by some external forces. Our electrostatic tests can be summarized by saying that, if comprehensive tests are run on each formulation prior to its processing, the hazard can be negated through grounding or other techniques. The point here is an important one and we recognize it—each separate formulation must be individually tested prior to processing so that complete and proper precautions may be taken. It is a safety problem which we believe can be satisfactorily handled once each formulation is adequately defined.

In summary, I would like to say that the Naval Propellant Plant has a proven continuous process for composite propellants, which essentially is a turbulent gas flow in lieu of mechanical mixing and contains "a substantial number of inherent safety and engineering advantages." There are no moving mechanical mixing parts, the process may be placed completely under remote control, and very small amounts of propellant are in mixing contact at any one time. The system is more flexible and less expensive, and results to date indicate comparable, if not somewhat better, finished products.

We recognize, of course, that pneumatic mix is no panacea, but we do feel it is a significant step in the state-of-the-art; and we, as well as private industry, will be constantly searching for cheaper, more efficient and safer methods for incorporating higher energy ingredients in solid propellant motors.

Mr. Jezek: I have several questions to ask. How do you people clean this mixer, let's say you use different formulations. After you mix the batch, how do you clean it?

Mr. Carsey: I'll go back and say one of the definite advantages of this system is by using our porous tube. We converge the liquids and solids so that there is a very minimum amount of impingement on the side of the porous tube or the cyclone separator. By our propellant and in the pilot plant propellants that we've run, we haven't had any problem cleaning this particular mixer. We just use the ordinary solvent cleaners on the cyclone separator and through the porous tube but I have a feeling that this would definitely be a problem if we were going into production. I can't personally answer the question of how you would clean a production unit. This would be a problem definitely.

Mr. Jezek: How much air do you use on this thing, what's the air pressure?

Mr. Carsey: We are forcing the air through, the only figure I have is a linear velocity of about 200 ft. per sec. in our tube and we are using about a 200 to 1, roughly, ratio between the gas and the air. We've only used air by the way, we've used no inert gas in our pilot plant.

Mr. Jezek: My last question. When you go to a production method use, do you intend to feed and screen continuously? In other words, you're not going to feed this system without screening are you?

Mr. Carsey: No, I don't think you could. Of course it depends on your oxidizer. One reason the industry has been able to go to continuous processing has been the fact that there has been a great deal of advance in the state of the art on being able to feed both solid and liquid materials. We're using a specially designed solid feeder for this particular work that we have been doing using by the way ammonium perchlorate. If you went to a production plant they would certainly have to be solved because any process has many engineering problems. One of them of course would be your solid to liquid feeders and the continuous feed of your material into your porous tube. This would be a very large problem and to date we have

been having very good luck with just a vibra screw, a solid feeder and a regular state of the art liquid feeder. Again we're dealing in pretty small amounts of material and we're actually running a continuous plant in a batch manner.

Mr. Mandano: What is the approximate length of that porous tube and what are your viscosity limitations, what viscosities can you handle in a mixing system of this nature? And do you have elaborate pressure control equipment to provide you with the proper type turbulence for adequate mixing?

Mr. Carsey: I'm not in a position to give answers to most of those questions that you asked. As far as viscosity is concerned the one propellant that we have used consisted of 75% oxidizer and 25% binder. It is a very high viscosity but I don't know the figure myself unless Dr. Dale happens to know it. But we feel that as far as viscosity is concerned, we're able to work with quite a viscous mixture using this system.

Mr. Mandano: You have side openings in your pipe for the control of the turbulence, now you are using a pressure to create the turbulent flow in your center tube. Do you have any equipment for controlling those pressures and is it very critical is the question?

Mr. Carsey: Yes, I'd say the critical part of this whole design from an engineering standpoint is this porous tube and the pressures that you've got there because this includes not only the pressures inside your tube but the way in which you feed your liquids into your solid stream. You've got to feed them perpendicular and you've got to feed them in such a way so that they actually will converge. I don't want to use the word slug but you've got propellant flowing down being mixed inside the center of the tube and there is virtually a boundary layer on the outside next to the porous tube because if you begin to beat off on the sides of your porous tube, you of course are creating problems that are a great deal more difficult to solve in your mixing so you must have critical pressures and you've got to have a critical feed system. All this, as I say, we've been very successful in making work with a pilot



plant operation and we feel it can work in a production standpoint. This is very definitely very critical and I don't know what pressure or I'm not intimately familiar with the system we're using on this. The whole secret of this thing is being able to converge this material down the middle of this porous tube with pressures coming in from the outside and turbulently mix it so that when you get to the end you have this homogeneous product.

Mr. Havron: I have two questions. What liquid to solid ratio do you expect to be able to get and how do you deaerate the propellant?

Mr. Carsey: I'll start out with the second question. First we have a cyclone separator which takes off a great deal of the air and then we actually put this thing through slit plates, I'm sure you're familiar with that and this was one of the things that really bothered us. When we first processed the grains we were extremely pleased to find out that we had no porosity problems and no void with it so we are doing an excellent job in our pilot plant of getting rid of the air. In reply to the first question. I can't really answer that. I feel and I think this is the stand, we should be able to process any of the composites. We feel that this process could be used for many of the composites currently being used today.

Mr. Diebold: You seemed to have worked only with the non-metallized formulations. Do you anticipate any problems working with the metallized formulations?

Mr. Carsey: We have only worked with the non-metallized formulations and we do foresee problems working with the metallized formulations.

Mr. Lusk: I was curious to know what level of hazard you might have placed on your mechanical feeders that you would have to use for this machine. You spoke of the vibra-screw and conventional methods.

Mr. Carsey: As I think I stated earlier, one of the problems you would face if you went to a production plant for instance with this process, would be your feeding

equipment, your solids and liquids feeders. Actually aside from the level of the problem, I really personally feel that probably your higher level of the problem has to do with cyclone separator, and its there where you're going to have to put a great deal of effort in it as far as the safety problems are concerned. The state of the art of solid and liquid feeders today both in this process and I'm sure you're familiar with quick mix and other processes which have been working, are pretty good and we think its currently such that we would be able to feed in our solid and liquid at least in the composite mixtures which are being made today without a great deal of problems. We foresee more safety problems and it is going to require more investigation and a great deal more care in the area of our cyclone separator and when you get down through this porous plate and you're actually trying to put the propellant into your propellant receptacle. I don't see that it is as big a problem as you have with other parts of the system but it depends a lot on your material, what you are trying to feed in. Certainly if we start to try to feed new materials into this process, we've got a whole series of new problems to solve.

Mr. Scannell: You say that you have a small amount of propellant in the porous pipe. If an incident were to occur wouldn't it propagate down to your finished product?

Mr. Carsey: Of course when you're talking about the possibility of building a production plant I think depending upon how much money somebody wants to spend to build one... how serious it would be and what it would be when it got down to the end whether you would actually have an explosion, detonation or simply a fire hazard is another question. But its certainly a valid problem and in the case of any large scale operation, would have to be very seriously considered. But like your pneumatic conveying process that Hercules showed today, propagation is always going to be a problem that you will have to worry about. The best thing you could say is that we just don't have anything happening inside there.

# GAP TEST CALIBRATION: A CRITICAL REVIEW

DONNA PRICE and IRVING JAFFE

U.S. Naval Ordnance Laboratory  
White Oak, Silver Spring, Maryland

**ABSTRACT:** A critical review of gap test calibrations made at NOL shows that film reading error can amount to 1 to 20% in the velocity-distance curve; each 1% error in the shock velocity is magnified 2.8 to 8 times by conversion to shock pressure. Smoothing procedures to minimize these errors are described.

Calibrations with various lots of tetryl and one lot of pentolite boosters are compared. An earlier interpretation of one set of data is reversed; pentolite and tetryl boosters are shown here to measure the same 50% pressure over a range of 20 to 120 kbar.

The inadequacy of the Hugoniot relation for Plexiglas (or any other solid) at low pressures is discussed and illustrated.

In the last four years, a number of calibrations of the standardized NOL gap test have been made at NOL. As a result, improved methods of reducing the data have been developed, and regions of relative uncertainty disclosed. It is the purpose of the present paper to review critically all the NOL calibration work, and to report the results obtained by use of the improved procedures. It also serves to correct an erroneous interpretation of data reported earlier.

The gap test calibration gives the pressure amplitude of the shock ( $P$ ), caused by loading Plexiglas with a standardized tetryl donor, as a function of the shock path in the Plexiglas.

Moreover, it is the relationship between shock pressure and attenuator gap thickness for the dimensions of the standardized test (1,2). The method of calibration is a familiar one; generally, the shock front of a tetryl loaded Plexiglas rod is followed by a smear camera (3). The resultant distance-time (t) record can be converted to shock velocity (U) vs distance (x). Combined with Hugoniot information for Plexiglas (U vs particle velocity, u, data) and the hydrodynamic relation

$$P = \rho_0 U u \quad \begin{array}{l} P \gg 1 \text{ bar} \\ \rho_0 = \text{density} \end{array} \quad (1)$$

the data can be converted to the desired P vs x form. Two variations on this method have also been used: replacement of a single rod with several of different lengths, and measurement of (a) both shock velocity and free-surface velocity ( $u_{fs} \sim 2 u$ ) or (b) shock velocity in Plexiglas and in water adjacent to the Plexiglas to obtain u by impedance relations.

The smear camera record showing x vs t of the shock front in plexiglas provides the basic data in each case. Our camera uses 35 mm film and has a writing speed of 1.3 mm/ $\mu$ sec. For a 100 mm long rod, we estimate that x can be read to 0.2 mm; t to 0.03  $\mu$ sec. The first 3.2 mm of shock path are obscured by the shield between the detonator and the rod; because of this and of optical disturbances, the earliest reading that can be taken is at about 5 mm and 1  $\mu$ sec. Consequently in the first (and steepest) portion of the x-t curve, reading errors of about 5% in each variable can be expected, whereas at the end of the curve (ca. 100 mm), errors will be of the order of magnitude of a few tenths of a percent. It is, however, the U vs x curve rather than the x-t curve that is required. Differentiation of the x-t curve to obtain the U-x curve can introduce errors in U greater than the sum of the errors in the corresponding values of x and t. As a

result, we estimate that errors in velocity may range from 20% at 6 mm/ $\mu$ sec to less than 1% at 3 mm/ $\mu$ sec.

Some averaging and smoothing of the data can minimize errors at small  $x$  and permit reproducible extrapolation into the 0 to 5 mm range where measurements cannot be made. In the earliest calibration (3), the  $x$ - $t$  curve (average of 5 shots) was smoothed graphically, differentiated graphically, and the resultant  $U$ - $x$  curve smoothed and extrapolated graphically. The present and preferred method utilized analytical procedures to smooth the graphically derived results. In other words, two independently (and graphically) derived  $U$ - $x$  curves are compared to  $U$ - $x$  curves obtained by an IBM routine of least squares fit of the raw data ( $x$ - $t$ ) to several analytical functions. The most successful smoothing of the graphical results in the range of 0 to 60 or 70 mm has been obtained by fitting data to the exponential function

$$x(t) = at + c - ce^{-mt} \quad (2)$$

where  $a$ ,  $c$ , and  $m$  are constants (see Ref. 4). Beyond this range, there are questions about the validity of both the  $U$ - $x$  and  $P$ - $x$  curves; some of these questions will be raised later.

The use of Eqn. (1) to convert  $U$ - $x$  data to the desired  $P$ - $x$  form will magnify any error already present. For example, if we use a Hugoniot in the form

$$U = A + Bu \quad A, B \text{ constants} \quad (3),$$

Eqn. (1) becomes

$$P = p_0 \frac{U(U-A)}{B} \quad (4)$$

For the Reference (3) values of  $A$  and  $B$ , it is easy to show that a 1% error in  $U$  will result in a 2.8 to 8.0% error in  $P$  as  $U$  varies from 6 to 3 mm/ $\mu$ sec. In other words, at high pressures where reading errors are high, the conversion error is relatively small; but at low pressures where reading errors are small, the conversion error is relatively large.

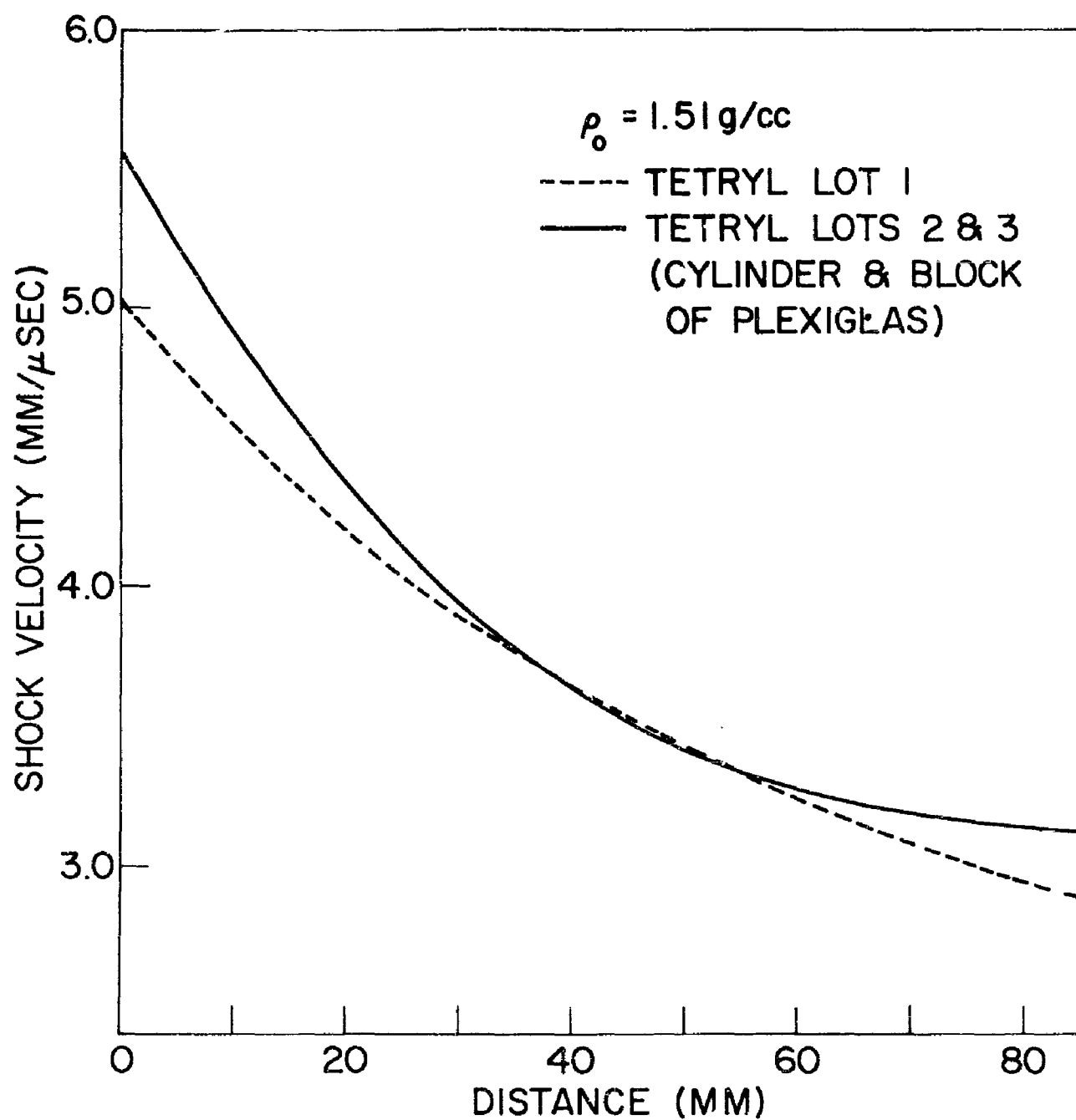


FIG. 1 SHOCK VELOCITY DISTANCE CURVES FOR TETRYL LOADED PLEXIGLAS

In comparing the different calibration curves, the conversion error can be ignored by working with the U-x curves. Fig. 1 shows curves obtained with three different lots of tetryl pellets (all of  $\rho_0 = 1.51$  g/cc). The lower dashed curve is for the first lot loading a Plexiglas rod (3); the solid curve is an average of three nearly coincident curves: tetryl lot 2 loading of a rod and of a block of square cross-section (edge length equal to diameter of the rod) and tetryl lot 3 loading of a rod [Calibration data for each of these will be reported (4,5).] It is evident that, although lots 2 and 3 are very similar, both differ significantly from lot 1. It is necessary, therefore, to calibrate every lot of pellets before using them in the test. The solid curve of Fig. 1 is essentially the calibration curve for the current NOL tetryl pellets.

In Fig. 2 this solid calibration curve for tetryl is compared with that obtained with pentolite (PETN/TNT, 50/50) pellets of  $\rho_0 = 1.56$ . The 50% gap value was also determined with tetryl and with pentolite loading for each of three explosives; for these explosives the gap range was 4-53 mm, i.e., within the x range covered by the calibration procedure. From Fig. 2 and Eqn. (4), the 50% shock velocities and 50% gap pressures were also determined. These same data were used to compare tetryl and pentolite donors in an earlier paper (6), but the interpretation of them then was based on the incorrect calibration curve for the tetryl used. The present results, shown in Table 1, reverse that earlier interpretation.

The data of Table 1 show that the different donors give the same 50% shock velocities within 2.5% of their mean value, and the same 50% pressure within  $\pm 10\%$ . In view of the errors to be expected in U and those of conversion to P, 10% cannot be considered an experimentally significant difference. In other words, within experimental error, tetryl and pentolite donors of these dimensions result in the same 50% pressures, and the pressure amplitude rather than the complete pressure-

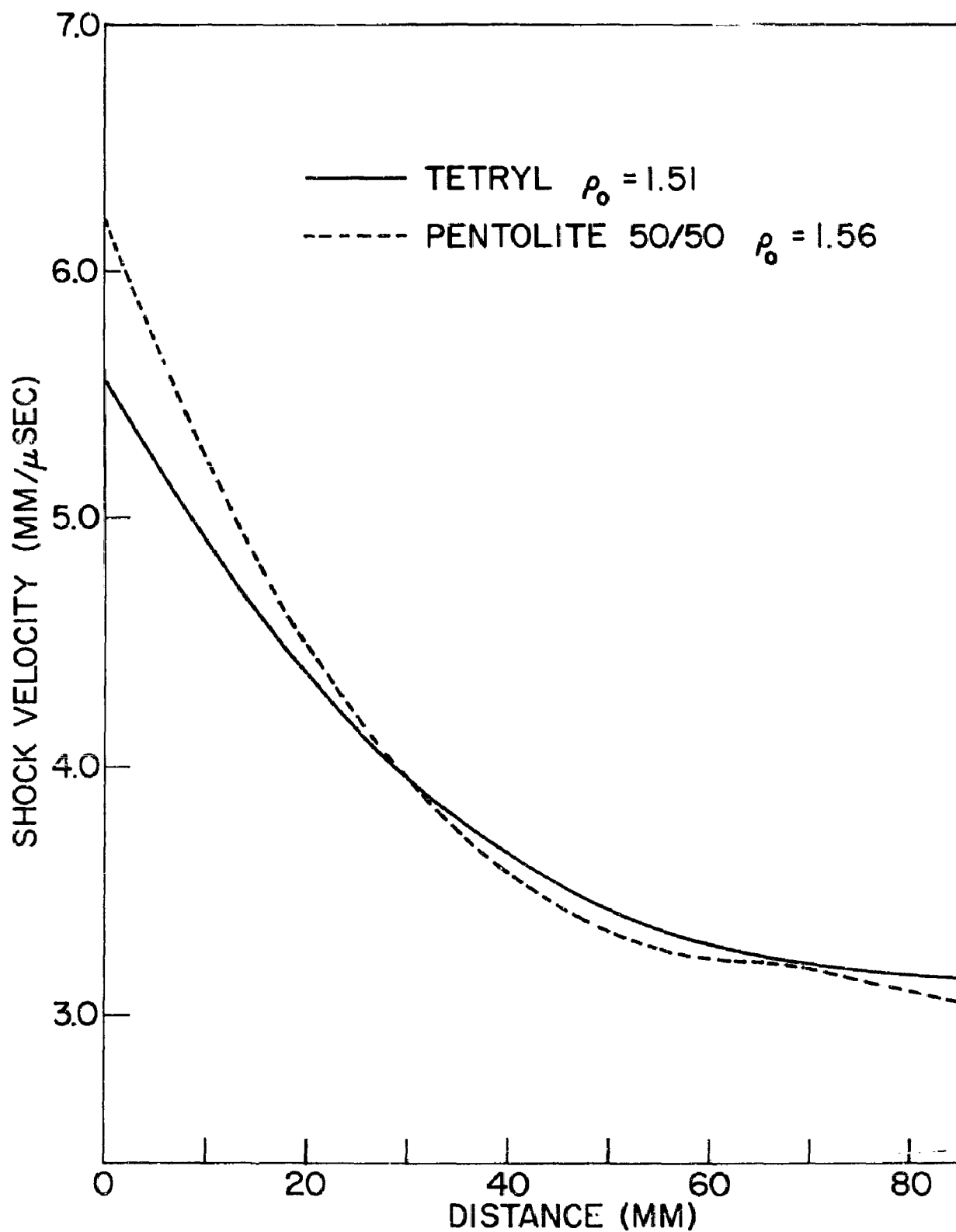


FIG. 2 SHOCK VELOCITY DISTANCE CURVES FOR EXPLOSIVELY LOADED PLEXIGLAS



TABLE 1

50% Pressure Determined with Two Different Donors

Test Material	Donor*	50% Values		Shock Velocity U, mm/ $\mu$ sec	$\Delta U$ , %	Pressure kbar	$\Delta P$ , %
		Gap, mm					
NQ/Wax, 95/5	T	4.06		5.310		112.7	
1.55 g/cc	P	6.35		$\frac{5.580}{5.445}$	$\pm 2.5$	$\frac{132.0}{122.3}$	$\pm 8$
NQ	T	11.7		4.815		83.7	
				$\frac{4.955}{4.885}$	$\pm 1.4$	$\frac{92.5}{88.1}$	$\pm 5$
Comp B-3	T	53.1		3.400		21.5	
				$\frac{3.290}{3.345}$	$\pm 1.7$	$\frac{17.7}{19.6}$	$\pm 10$

\* T: Teteryl pellets Nb 1878-96, 1.51 g/cc.

P: Pentolite 50-50 pellets, 1.56 g/cc.

time history of the pulse seems to determine the threshold stimulus required to initiate detonation. This may mean merely that the explosives are so similar that in the same dimensions they produce approximately the same pressure-time history. Alternatively, the stress pulses produced may be of such a shape that their effects are independent of time. In the case of tetryl we now know that the pressure falls from its maximum value to zero in 2  $\mu$ sec or less (4).

The first area of uncertainty we have sketched is then the value of the pressure measured in gaps of up to 70 mm length. This uncertainty can be reduced with better instrumentation and measurements. The second problem I am going to raise is perhaps more serious, and that is the validity of the hydrodynamic pressure concept at large gap thicknesses,  $x > 70$  mm.

We know that there are a number of sets of Hugoniot data for Plexiglas. To illustrate some of the differences, I'll take two from the NOL work:

$$U = 2.588 + 1.514 u \quad (5)$$

from work of Jaffe et al. (3) and

$$U = 2.710 + 1.568 u - 0.307 u^2 \quad (6)$$

derived by Coleburn(7).

Used with Eqn. (1), these give

$\frac{U}{\text{mm}/\mu\text{sec}}$	$P(\text{kbar})$	
	Eqn (5)	Eqn (6)
5.59	130.8	126.9
3.00	9.6	6.6

At higher pressures, the percent difference in P is small, but at lower ones, it is enormous. In an attempt to obtain a more reliable value, Liddiard (5) has been measuring free surface velocity as well as shock velocities. At  $x = 130$  mm,  $U = 3$  mm/ $\mu$ sec, he gets via Eqn. (1), a pressure value of

3.5 kbar, suggesting that the low pressure values from Eqns (5) and (6) are too high by factors of 2.7 and 1.9, respectively. Moreover, Liddiard's results using thin films on the Plexiglas free surface suggest (1) that the U-u curve does not extrapolate linearly\* and (2) that the rigidity of the material is affecting the free surface velocity. We are probably in a region where the hydrodynamic approximation is invalid, and where shear and tension are quite as important as compression. This is a region of great practical importance because it contains the most sensitive materials. Unfortunately, we may not be able to characterize it adequately by a hydrodynamic pressure - or by that pressure characteristic of an infinite system. In summary then:

1. The calibration curves of pressure vs gap length are subject to errors of reading the records, differentiating the distance-time curve, and converting velocity to pressure.

2. With the present techniques and equations of state, pressure values are believed to be good to 10% up to gap thickness of 60 mm.

3. Each lot of tetryl pellets must be calibrated before use in the standardized test. Up to 60 mm gaps, tetryl and pentolite pellets measure the same initiating pressures.

4. At gap thicknesses greater than 60 mm (pressures of about 20 kbar) the current Hugoniot for Plexiglas overestimate the pressure. This may result from a dimensional effect in the test with consequent failure of the 1-D Hugoniot.

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\*It is still possible that the linear curve holds for a true one-dimensional situation.

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2. D. Price and I. Jaffe, AIAA Journal 1, 389-94 (1963).
3. I. Jaffe, R. Beauregard, and A. Amster, ARS Journal 32, 22-25 (1962).
4. I. Jaffe, J. Toscano, and D. Price, NOLTR 64-66, in press.
5. T. P. Liddiard, Jr., NOL, unreported data.
6. J. Toscano, I. Jaffe, and G. Roberson, AIAA Journal 1, 964-66 (1963).
7. S. J. Jacobs, T. P. Liddiard, Jr., and B. E. Drimmer, Ninth Symposium (International) on Combustion, Academic Press, New York (1963) pp 517-34. Quotes Coleburn's results originally given in NavWebs Report 6026 (31 Oct 1960).

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Dr. Dale: Do you have any information on the variation of pentolite densities and K bars that you might get from that?

Dr. Price: No, I'm sorry, these particular pellets were prepared at NOL to very rigid specs and we did not vary the range and perforations nor the density. They were all, incidentally, made at a density of 1.56 grams per cc and the variations from that was less than 1%. It was an experimental lot in contrast to the tetryl pellets which were not involved in experimental lot.

## HAZARD CLASSIFICATION TESTING OF SOLID PROPELLANTS

Gerald Couch  
United Technology Center  
Sunnyvale, Calif.

The purpose of performing hazard classification testing of solid propellants is to obtain information for determining the ICC classification and the Military, or Department of Defense, classification. The ICC classification will dictate the method of packaging, labeling, and shipping of the propellant, or devices containing the propellant. The military classification will dictate the design and siting of facilities and operating, or handling procedures. Either or both classifications may influence state and other local rules or regulations.

More basically, the tests are designed to determine whether or not a propellant will detonate, and, if it does, under what conditions it will detonate and what its relative sensitivity is.

One often hears the term "detonation" used very loosely, even among safety people. This loose use of the term can lead to considerable misunderstanding among those who may have to make major decisions in developing safety criteria. A detonation as used in this presentation refers to a chemical reaction which proceeds through the reacting material toward the unreacted at a supersonic velocity. Gas and energy are released at a very rapid rate, at a rate sufficient to form a pressure, or blast wave, which moves away from the reacting material at supersonic velocities.

The currently accepted test procedure for determining the classification of propellants is contained in Section III of Air Force Technical Order 11A-1-40 (TB 700-2, BuWeps Inst 8020.3). It is proposed to present in this paper the results obtained in performing the tests on typical PBAA type of composite propellant using aluminum as a fuel and ammonium perchlorate as an oxidizer.

Since the propellant was available in sufficient quantity to do so, the first test performed was detonation test. A two inch (2") cube of propellant, to which was attached a No. 8 blasting cap, was placed on top of a 1.5 inch diameter by four inch (4") high lead cylinder. Failure of the cylinders to mushroom, or be flattened, is indication the propellant did not detonate. In the absence of a detonation, the propellant is subjected to a more

severe test by using Engineer's Special Blasting Cap. Slide I shows the lead cylinders after a test. Note the absence of mushrooming. Failure of detonation to occur with this test negates the need to perform an impact machine test which is of doubtful value anyway.

An ignition and unconfined burning test is performed by placing a one inch (1") cube of the propellant on kerosene-soaked sawdust which is remotely ignited. No witness device is necessary since detonation of the propellant can be detected by a sound report and significant cratering at the point of detonation. This test is followed by igniting in the same manner a two inch (2") cube. This test is repeated and then followed by igniting four (4) two inch (2") samples placed end-to-end in a single file. No detonation occurred with the tested propellant.

A Thermal Stability test is performed in the laboratory to determine if the propellant will ignite or unduly expand at maximum anticipated storage temperatures. A sample of propellant was maintained at a temperature of  $78^{\circ}\text{C} \pm 3^{\circ}$  in an oven for a period of 48 hours. The propellant neither ignited nor underwent any visible change during the test.

A Differential Thermal Analysis test is a common laboratory test to determine the temperature, under the particular test condition, at which the propellant ignites, explodes, or decomposes. In this test it ignited at  $500^{\circ}\text{F}$ . The main value of this test is to allow a comparison with the ignition temperature of other more familiar items obtained in the same manner.

The completion of these tests without the occurrence of a detonation permits one to conclude that the propellant can be classified as Class B under the ICC Regulations. The propellant, for shipping purpose would be described as a Propellant Explosive (Solid), Class B. For military handling purposes it can be designated as a Class 2 Fire Hazard, unless it is confined.

The next series of tests is intended to determine the effects of confinement and the relative sensitivity to detonation of the propellant.

This slide (Slide II) shows the test set-up for a four inch (4") critical diameter test. The test sample consists of a solid grain cast in a four inch (4") I.D. steel pipe with a 3/8 inch thick wall. This was set on a 4x12 inch cone of pentolite. A witness plate of 1/2 inch steel was placed atop the sample. The pentolite cone was initiated at its apex with an Engineer's



6



7



8



9



10

J-2

Blasting Cap



1



2



3



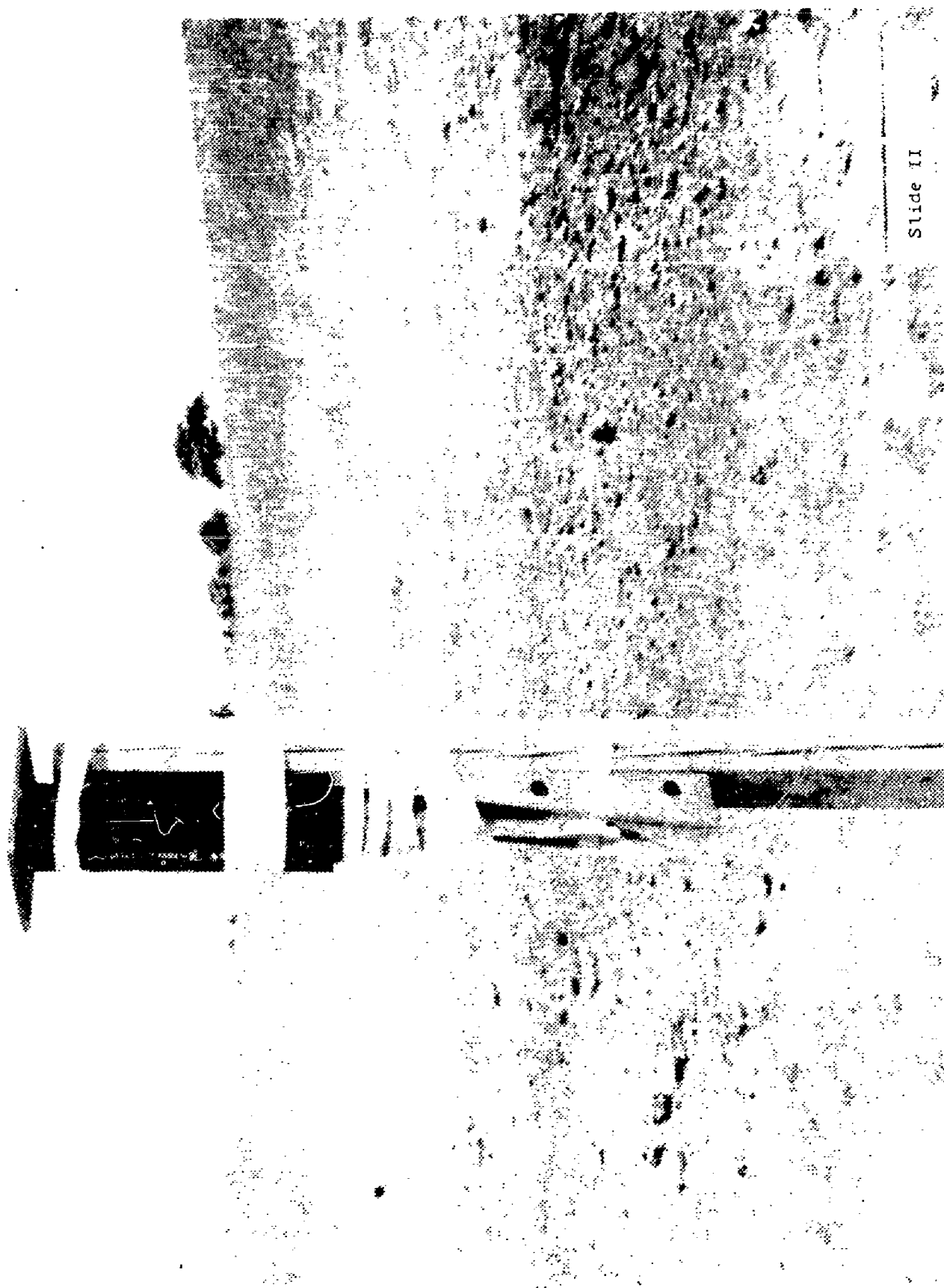
4



5

#8 Blasting Cap

Slide I



Slide II



Special Electric Blasting Cap. Slide III shows the effect of the test on the witness plate. Failure to penetrate the witness plate is evidence that the propellant did not detonate.

An eight inch (8") critical diameter test is similarly performed. To assure against loss of the witness plate the test should be performed with the witness plate next to the ground as shown in Slide IV. Another advantage of this arrangement forces the fragments of the grain and case in a downward trajectory. Note that blast gages are added to this test to measure the overpressure. Since blast, or overpressure, measurements are to be taken, it is necessary to calibrate the test configuration by using, in an otherwise identical test, an inert propellant with a density similar to that of the propellant to be tested. Although it was obvious that the propellant contributed to an increased overpressure and an analysis of the blast gage measurements indicated it was approximately a 25% TNT equivalent, even the empirical value of such a test is doubtful. Doubts arise because only one test is performed, there is no information on which to scale up from the sample quantity, and undefined test conditions, such as the height of the test above the ground and the type of terrain over which the test is performed can affect the results.

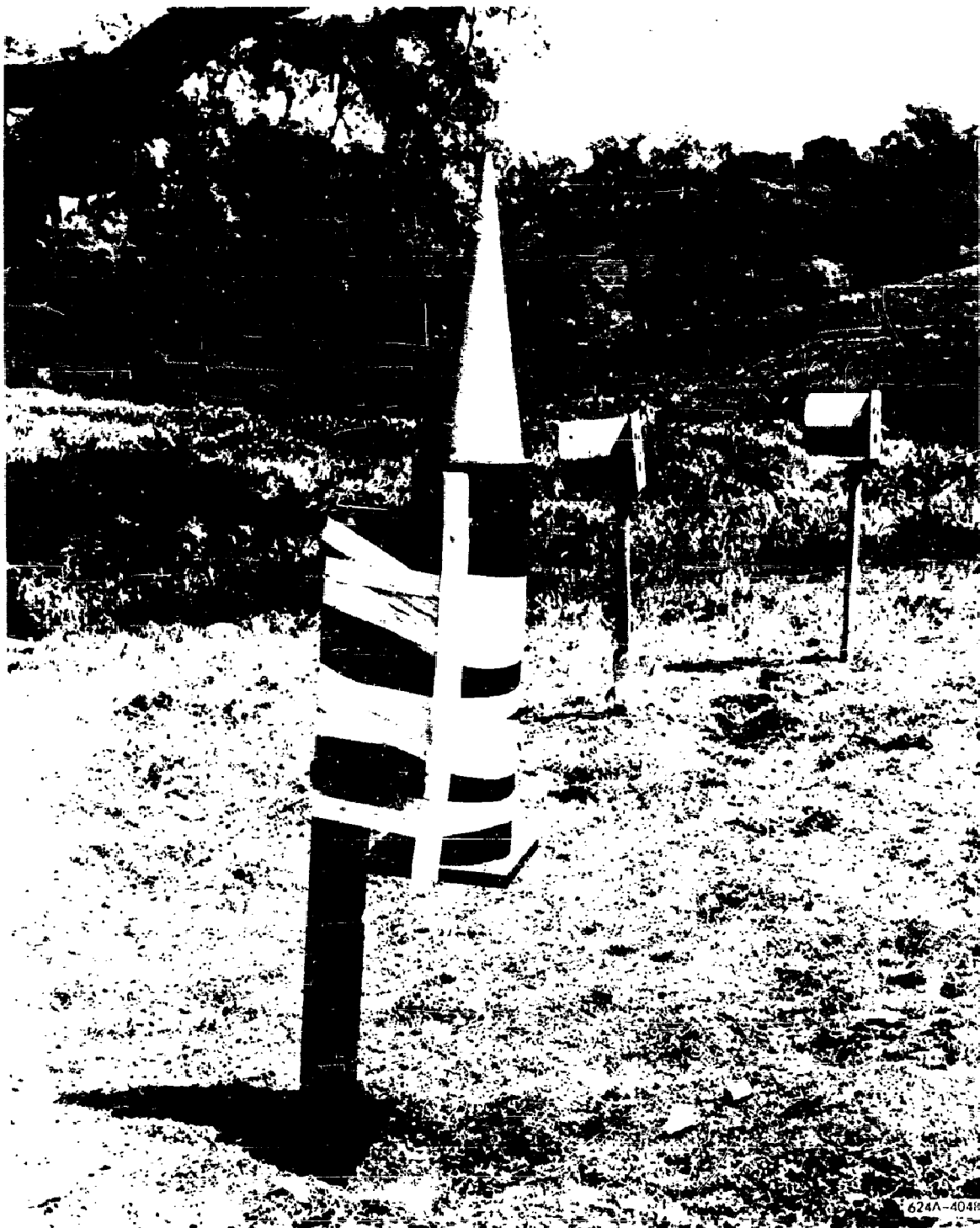
Slide V depicts the test set-up for the performance of card gap tests to determine the relative sensitivity of detonable propellant. Although some consideration has been given to deleting this test if the critical diameter tests do not produce a detonation, the difference in the test configurations has led to some concluding that a propellant may detonate with a zero card gap test, even though it does not detonate in the critical diameter tests. A test with zero cards on the propellant tested did not produce a detonation.

An external heat test using a bonfire is performed on a simulated five inch (5") diameter motor as shown on Slide VI. Temperature sensing devices were placed at eight foot (8') intervals in the direction of, and four feet (4') below, the line of the exhaust. The temperature at eight feet (8') in one test was between 150° - 200° F, in another 400° - 500° F. Beyond eight feet (8') the temperature was less than 150° F. It is believed the devices recorded the temperature of the bonfire rather than the propellant exhaust. This is unavoidable with the prescribed test configuration. The motors after ignition burned normally.

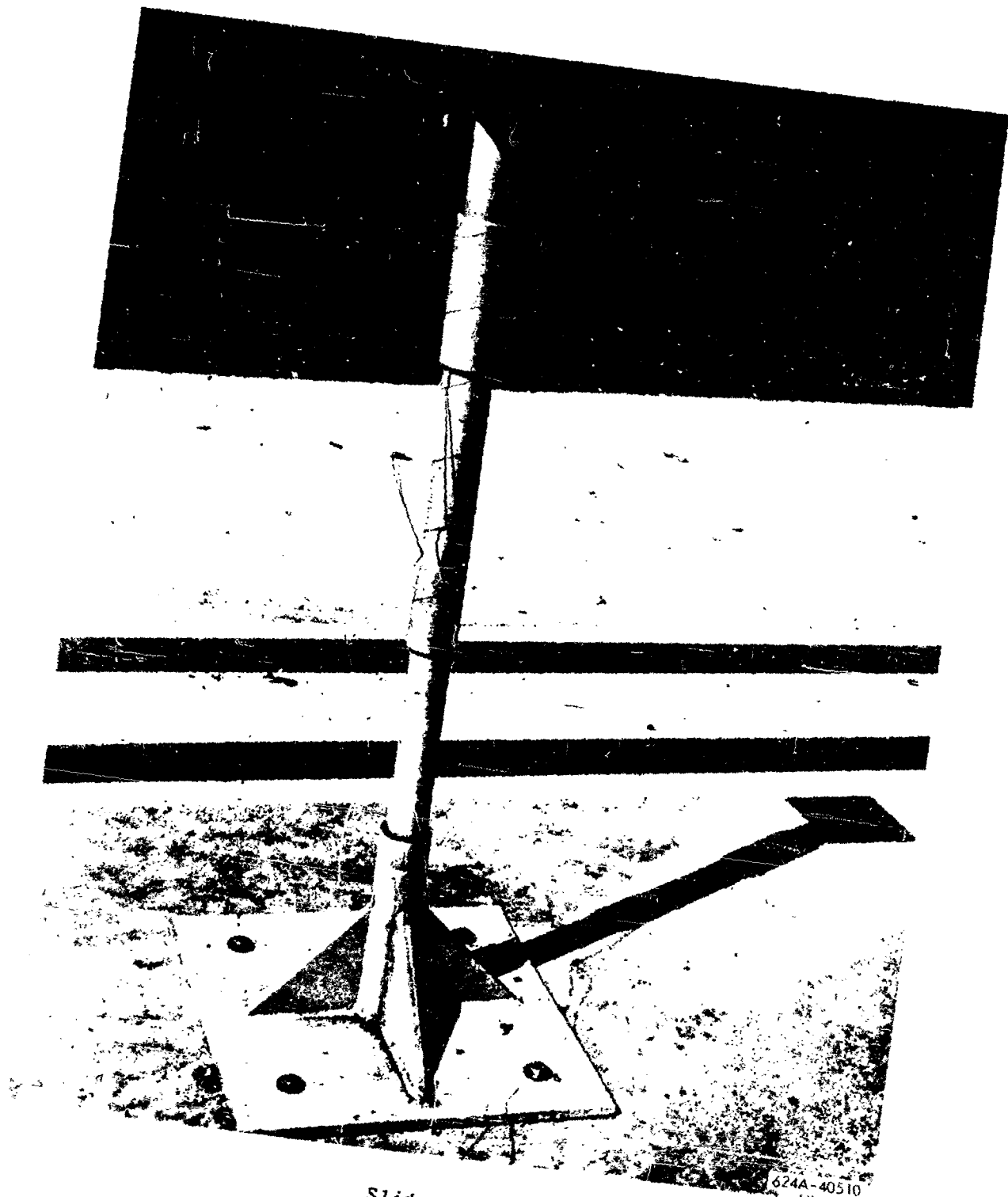
Slide VII shows the effect of bullet impact upon the simulated five inch (5") diameter motors using a fifty (50) caliber armor piercing projectile. An expected increase in burning of the fragmented propellant over-pressurized the motor and caused the aft end to blow out. The unfragmented motor case is indication of no detonation. This was also confirmed by motion pictures.



Slide III



Slide IV

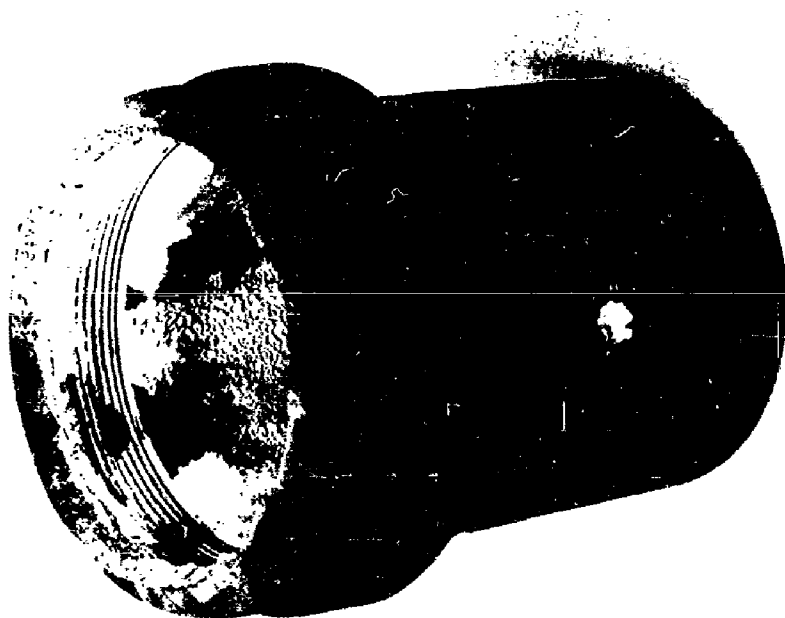
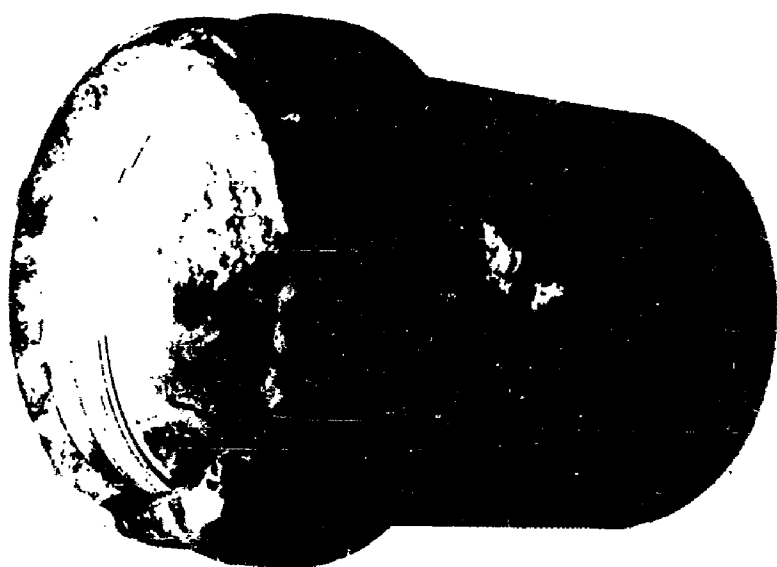


Slide v

624A-40510



Slide VI



Slide VII

These tests demonstrated that the propellant tested does not produce a detonation hazard under foreseeable handling conditions. It is believed that any composite PBAA type propellant with ammonium perchlorate oxidizer will give similar results.

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Unidentified: What was the distance and velocity of the bullet impact test?

Mr. Couch: The distance was 100 ft. as specified in T.O. 11A-1-47.

Mr. Cook: What were the dimensions of the TNT cones on the 4" and 8" test?

Mr. Couch: They were pentolite, 4" and 8" diameter respectively and the length was three times that respectively.

Mr. Oeinck: In the bullet test with the .50 caliber, was the steel the same type of steel that you use in your large solid motors?

Mr. Couch: No, mild carbon steel was used in the test motors.

Mr. Oeinck: One more question, on the thermal stability test, you said 78° Fahrenheit or Centigrade?

Mr. Couch: Centigrade as specified in T.O. 11A-1-47.

Dr. Dale: What kind of blast gage did you use for this low over-pressure measurement?

Mr. Couch: Bikini type gages and we don't find them to be very good.

Dr. Dale: Have you tried any BRL gages or any of the ARC gages?

Mr. Couch: On these particular type propellants we have not, but we plan to.

## TITAN III SOLID MOTOR IMPACT TEST

F. H. Weals  
U. S. Naval Ordnance Test Station  
China Lake, Calif.

This presentation will describe an impact test of a large solid propellant motor simulating many of the characteristics of stage zero of TITAN III. The test was conducted at the request of the Space Systems Division of the Air Force Systems Command. The test date was 13 June 1964 and the test site was the SNORT track of the Naval Ordnance Test Station, China Lake, California.

The experiment simulated the impacting of a burning, zero stage motor on a hardened surface - a situation which might occur in the event of a vehicle malfunction in the early portion of flight. The planned impact velocities represented one severe condition of fallback.

### Movie Narration

The motor was transported by truck from United Technology Center at Sunnyvale, California. It was shipped in three major pieces; the forward closure with igniter; the cylindrical center segment, and the aft closure with the nozzle installed.

The motor was assembled in the vertical, nozzle-up position on a specially constructed assembly stand. The assembly included installation of fore and aft sled rings which attached to the motor and served as motor supports during rotation from the vertical to horizontal position and transmitted the load from the motor to the sled trucks. The test motor was 10 feet in diameter and weighed 128,000 pounds of which 92,000 pounds was propellant. The length was approximately 16 feet excluding the nozzle. The test item varied from flight hardware in having a heavier motor case consisting of 3/4" steel. Additionally, it had only one 10 foot long cylindrical center segment in lieu of five (5) and was, therefore, considerably shorter than a stage zero motor for TITAN III.

The motor and sled rings were assembled in the following order:

1. Forward sled ring
2. Forward motor closure
3. Cylindrical center segment
4. Aft closure with nozzle
5. The aft sled ring



Two cranes, each of 90 ton capacity, were provided by contract and were employed in the assembly and rotation procedure. Rotation from the vertical to horizontal position proceeded smoothly.

The motor was placed on a sled consisting of the two (2) support rings, four (4) trucks and 14 isolation skids - 3 skids on each of the forward trucks and four (4) skids on each of the rear trucks.

The target face was constructed of reinforced concrete blocks. The total concrete impact surface was 20 feet square and six (6) feet thick. The impact surface had openings to accept the sled trucks and allow the motor to experience the initial impact. The concrete face was backed by a large steel plate three (3) inches thick and this, in turn, by earth fill approximately 35 feet in the direction of motor travel.

The 4000 foot run from ignition to impact required 13.31 sec. Chamber pressure was 550 psi. The acceleration curve was essentially flat with an average value of 1.4 G's. The motor impacted at a velocity of 667 feet per second with approximately 800,000,000 ft. lb. of kinetic energy.

The nozzle remained in place at the impact site. The forward closure was doubled over and came to rest 270 feet down range. Concrete from the target was scattered from 0 to 2300 feet. The 3" steel plates behind the target were not penetrated but suffered distortion.

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Figure 1 shows the gage layout for recording pressure-time history. BRL self-recording gages and Kistler Piezo-Electric gages were used.

Note the position of gage lines relative to the target. The North Leg (Leg #1) was sheltered by the target, whereas the closer stations on the Southeast Leg (Leg #3) may have sensed reflections from the target.

Position "A" was at 91 ft. and position "F" was at 1500 ft. from the target.

# PRESSURE GAGE LOCATIONS

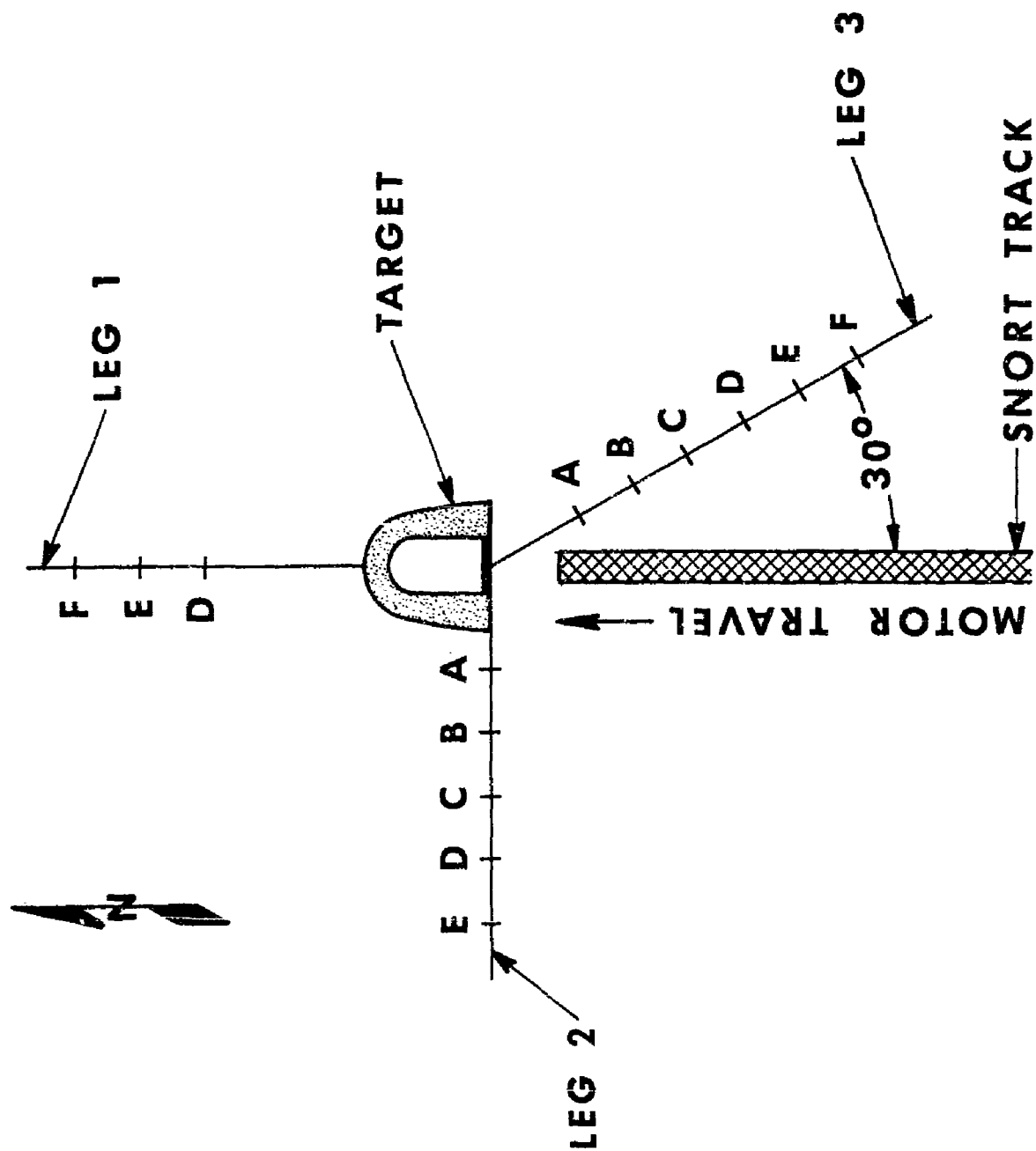


Figure 1

Figure 2 shows the plotted positions of peak overpressure data recorded by the gages.

Two reference lines are also shown - one for 4,000 pounds and the other for 16,000 pounds of TNT.

The reference lines were derived from BRL Memo report No. 1518 dated April 1964.

Data points from the North, or sheltered, gage line tend to run near the 4,000 pound reference, whereas those from close-in positions on the southeast leg, at 30° with track, tend to run higher than the average.

Overpressure values at 1500 ft. were under 0.5 psi.

If TNT equivalent weights are computed for each point, the average of these weights approximates 7,000 pounds of TNT.

Similar averaging of what are considered to be the most representative points i.e., the mid-position and distant locations on Legs #2 and #3 shows a similar value.

This equates to 8.5% TNT equivalency. The theoretical and practical hazards in the use of the term TNT Equivalency are acknowledged.

Figure 3 shows plotted positions of impulse data. Again there is a trend of the north leg data to run low - the southeast to run high at close-in positions - and the west leg data to fall in between.

It can be seen that a line midway between the 4,000 pound and 16,000 pound references would be a reasonable fit. This midway position corresponds to 8,000 pounds of TNT or an equivalency of about 9.5%.

I want to emphasize that these values of overpressure and impulse are tentative. Data is still under review by BRL and some points may change. Additionally, the results cited will be given one or more independent technical reviews before a final report is issued.

# OVERPRESSURE vs DISTANCE

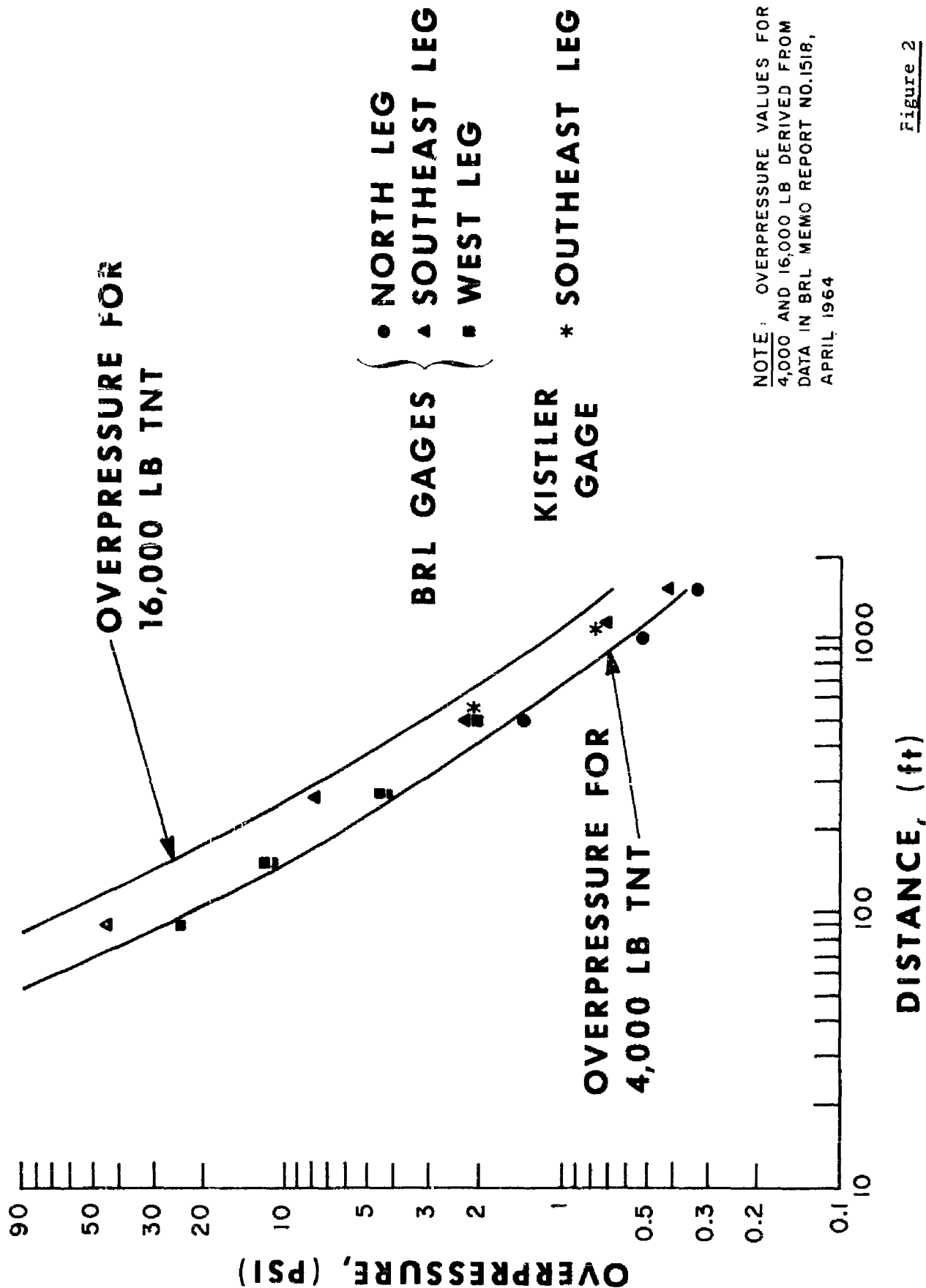
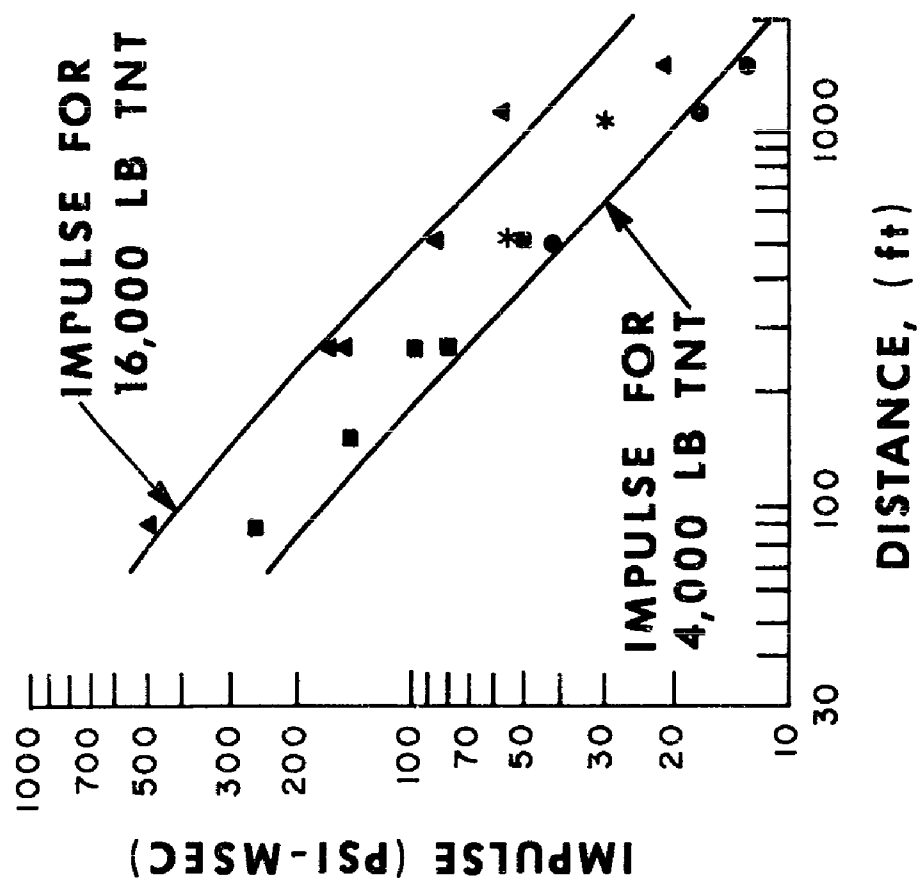


Figure 2

# IMPULSE vs DISTANCE



NOTE: IMPULSE VALUES FOR 4,000  
AND 16,000 LB DERIVED FROM DATA  
IN BRL MEMO REPORT NO. 1410, JUNE 1962

Figure 3

Figure 4 shows the maximum travel of fragments.

For the most part the distances shown represent the travel of isolated fragments and not multiple fragments. Burning propellant was thrown a maximum distance of 3,000 feet to the east and 2,850 feet to the west.

Unburned propellant is not shown here, however, its spread was relatively limited and the fragments were quite small. One large motor case fragment 6 ft. x 8 ft. traveled 2500 ft.

I wish to emphasize that the limits of travel shown here represent, for the most part, individual isolated fragments and not groups of fragments.

The application of the results of this test is beyond the scope of this presentation, however, I do wish to make one comparison. Overpressures of 0.5 psi occurred at less than 1500 ft. The 1.0 psi region approximates 800 ft. Maximum fragment travel is much greater than either of these overpressure distances.

In conclusion, I wish to mention some of the various organizations contributing to the planning and execution.

First, the Space Systems Division who provided overall guidance and the all-important money; and also the Aerospace Corporation working closely with SSD.

The United Technology Center provided the motor, some associated equipment and technical liaison.

The Air Force Rocket Propulsion Laboratory at Edwards AFB provided consulting services on overpressure, measurement and some gages.

The Coast and Geodetic Survey instrumented the NOTS Range area with seismic gear.

# MAXIMUM FRAGMENT TRAVEL

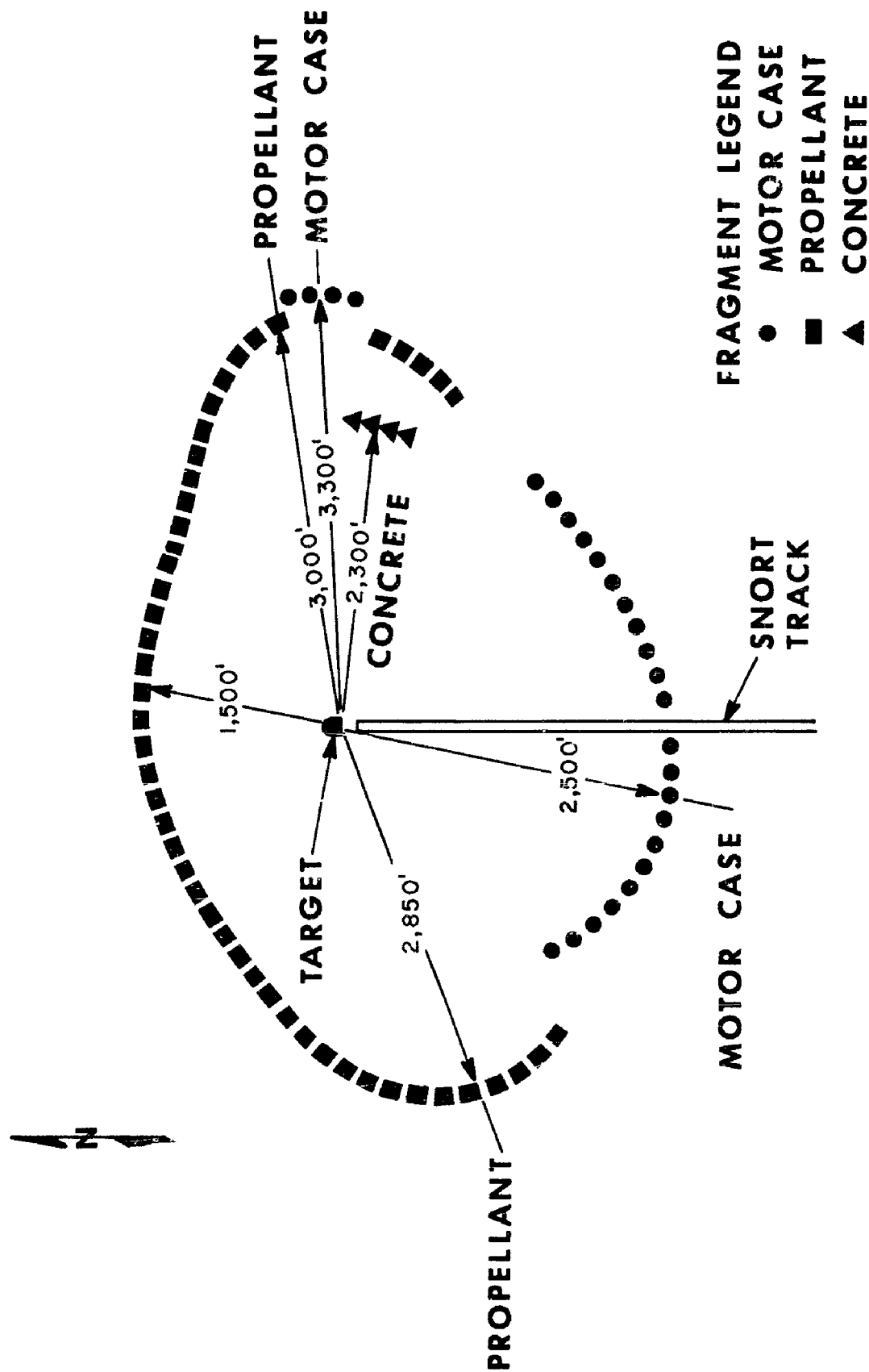


Figure 4

Mr. Jezek: Did you have any crater from this?

Mr. Weals: Yes, there was a crater, it was about 7 ft. deep. Of course this was impacting a vertical target, it wasn't impacting horizontally.

Mr. King: Did I miss the figures, or did you give the figures as to how much propellant was left by weight when it hit the pad?

Mr. Weals: It was 82,000 pounds.

Mr. King: From a total of 93,000 at the beginning?

Mr. Weals: We started out with 92,000 at ignition. This was a calculated figure of course, it was calculated to be 82,000 at impact.

Mr. Jenkins: Would it be possible to hazard a guess where this type of propellant would fit in to the new quantity-distance tables, Class 4, 5 or 6?

Mr. Weals: I don't think I would want to hazard a guess.

Dr. Shuey: This is idle curiosity, my eye seemed to catch a capacitive discharge spark from the head end of that motor just before it hit the steel plate in two of your shots. Is that what it is?

Mr. Weals: I think when steel hits concrete at this velocity you almost always get a flash.

Dr. Shuey: My question was, it looked like it was before it hit, I was curious if anyone had analyzed that.

Mr. Weals: It may be because of lack of definition in the picture. The igniter of course is up front and it may be a forward projectile, perhaps a bolt or probe of some kind that hit first, that is my opinion.

Mr. Jezek: I just want to know if these things are going to go up or down a highway when they take off?

Mr. Weals: It was shipped in the same way it was assembled, i.e., the individual components of the motor. By that I mean that it was in sort of an inverted position with



the nozzle up as you noticed. That meant that the forward closure, the steel plate, the convex surface of the forward closure was down and the exposed propellant was up. It was shipped with the igniter. The rear closure with the nozzle was shipped with the nozzle up.

Mr. Hart: I wonder if you could tell me first in review, this was a PBAA type propellant, correct?

Mr. Weals: Yes.

Mr. Hart: Could you indicate what was the oxidizer content and what was the total solid content?

Mr. Weals: I would like to refer that question to the UTC representative.

Mr. Couch: I'll give you only a round figure of the oxidizer being around 67% by weight.

## SAFETY CRITERIA IN THE DESIGN OF A CONE-VERTICAL PROPELLANT MIXER

Wm. Arbiter, Atlantic Research Corp., Alexandria, Va.

### ABSTRACT

The design of cone-vertical mixers currently being manufactured by Atlantic Research Corporation has drawn heavily upon the safety recommendations resulting from the Symposium\* at U. S. Naval Propellant Plant of Indian Head, Maryland, September 1961.

The cone-vertical mixer concept has inherent in it many important safety features not found in other mixers. Foremost among these are:

- 1) Adjustable blade-to-blade and blade-to-wall clearance,
- 2) No gears or bearings exposed to mix environment,
- 3) Minimal weight of components above mixing chamber,
- 4) Temperature sensing device located within the mix and with adjustable depth capability,
- 5) Liquid additions through blade shaft,
- 6) Large ratio of exposed surface area to volume, and
- 7) Variable peripheral blade speed at constant rpm as a function of depth in the mix.

These and other safety features are discussed and illustrated in the presentation.

Other technical features of these mixers, such as their ability to handle mixes of extremely high viscosity (up to 40,000 poise), their high mixing efficiency, their susceptibility to remote operation over a complete cycle (loading, mixing, discharging and cleaning) are also discussed and illustrated.

\* Wesche, O. A. and Browning, J. C. "The Function of the Vertical Mixer in Solid Propellant Processing - A Symposium".

## I. INTRODUCTION

### A. Atlantic Research Corporation Background

Atlantic Research Corporation has been involved in the research, development, and production of solid propellants and solid propellant rockets during all of its fifteen years of existence. Atlantic Research Corporation has been a leader in the development of high-energy propellants such as the aluminum-bearing formulations that made the Polaris fleet ballistic missile a reality. With a production capability of 5,000,000 pounds of solid propellant per year and as the producer of several types of rocket vehicles, numerous sizes of meteorological sounding rockets, a wide variety of auxiliary and control rockets, and several military rockets, ARC has acquired an enviable safety record. This has been accomplished only with considerable attention to safety engineering.

### B. Development of Test Equipment and Laboratory Mixers

Before a propellant can be handled with safety, its behavior must be predictable. Therefore as a first step in characterizing the various propellants with which they have worked, ARC personnel have been concerned with the determination of pertinent physical, chemical and mechanical properties and with controlling the processing procedures so to maintain the properties within such limits that predictable performance behavior is obtained. Where satisfactory instrumentation was not available ARC scientists and engineers developed such instrumentation. Several of these instruments are shown in the following figures. (Figures 1-3).

In addition to the need for better instruments for determining solid propellant properties, it was found that mixing equipment was inadequate, especially in laboratory sizes. Available mixers were extremely inefficient, requiring extended mixing periods for complete mixing. Since the probability of an incident occurring is bound to decrease if the time available for it to occur decreases, mixer designs were sought which could reduce the time

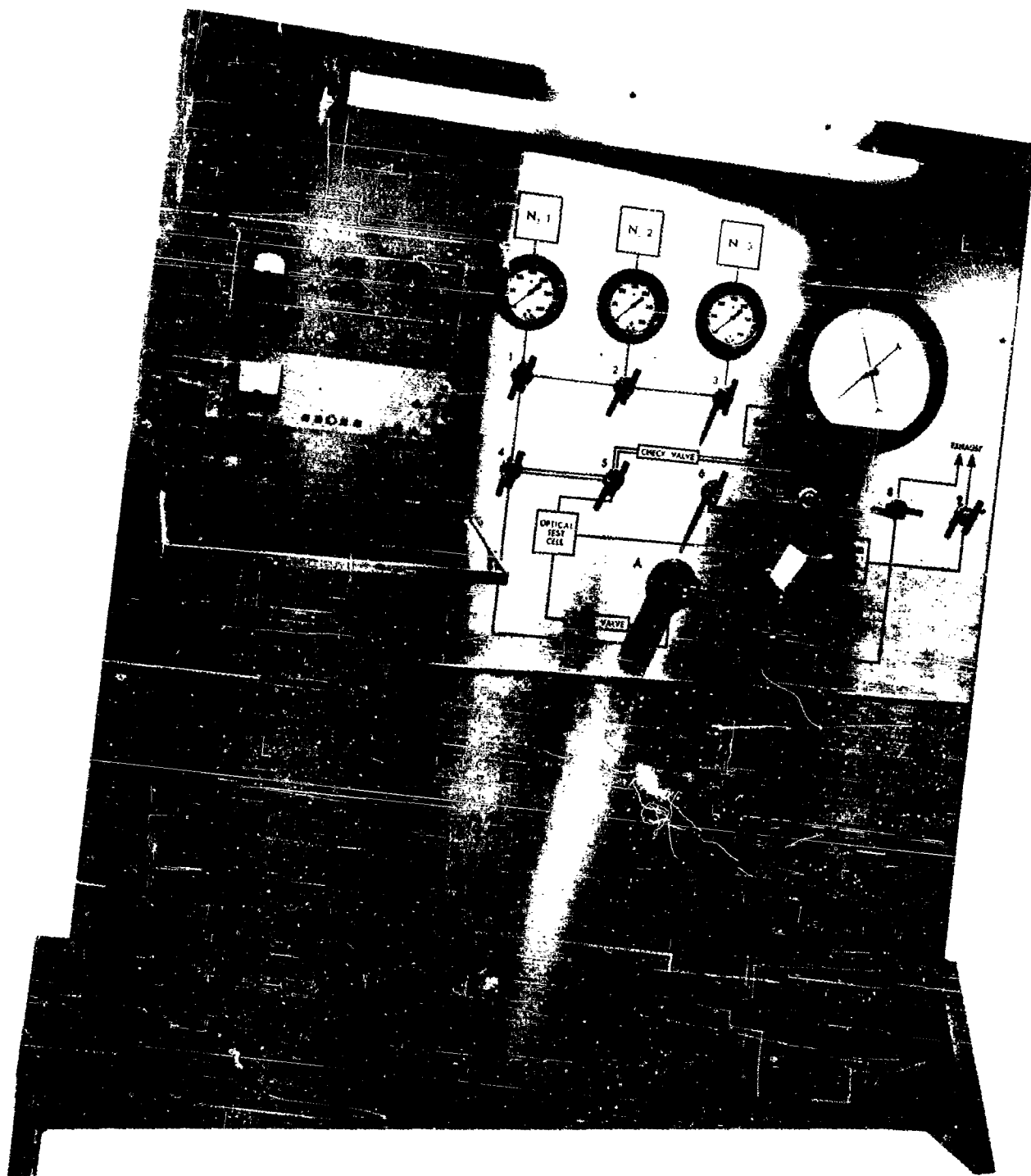


Figure 1. Safety and Control Panel for Strand Burner-Optical Bomb Combination System.

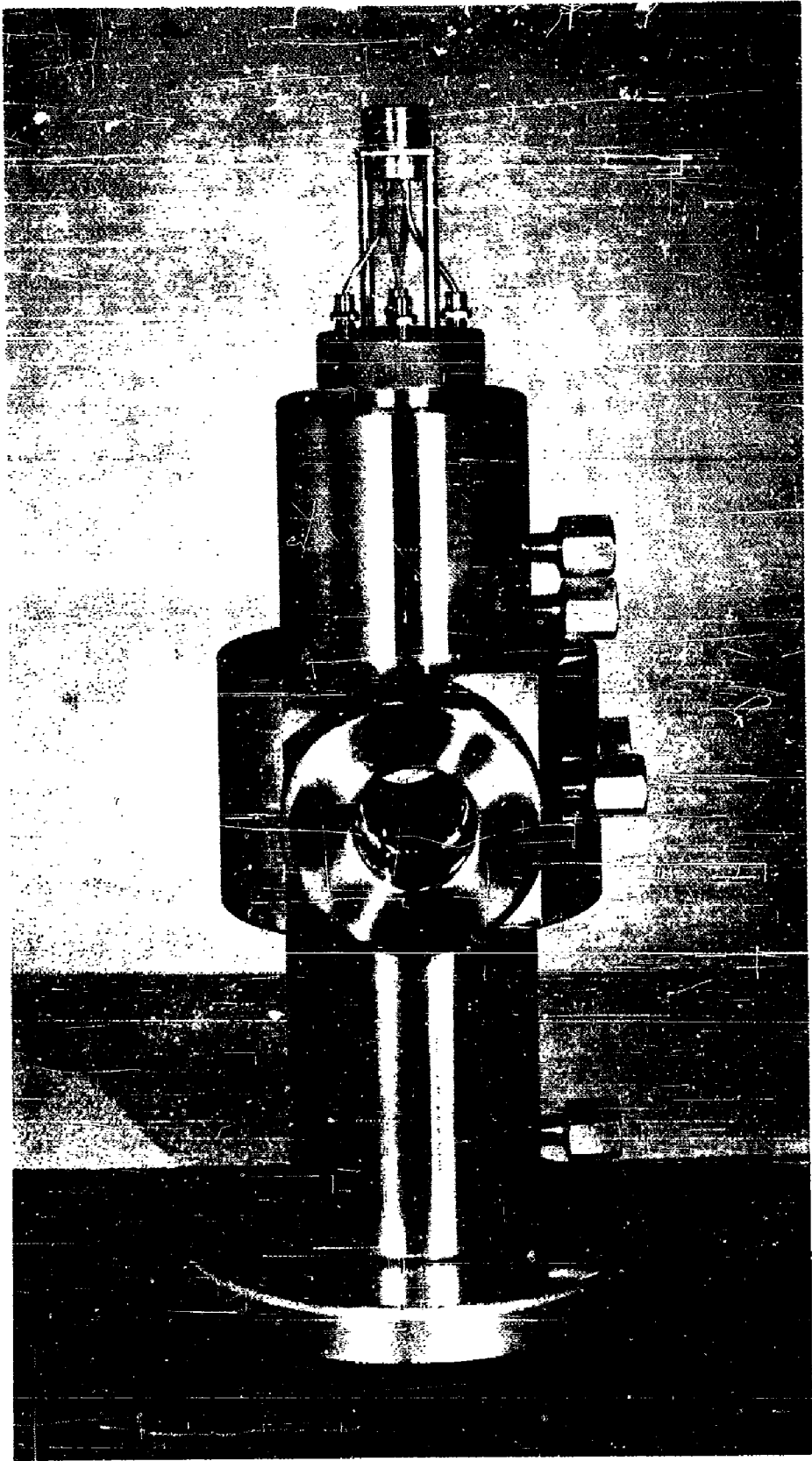


Figure 2. Optical Bomb.

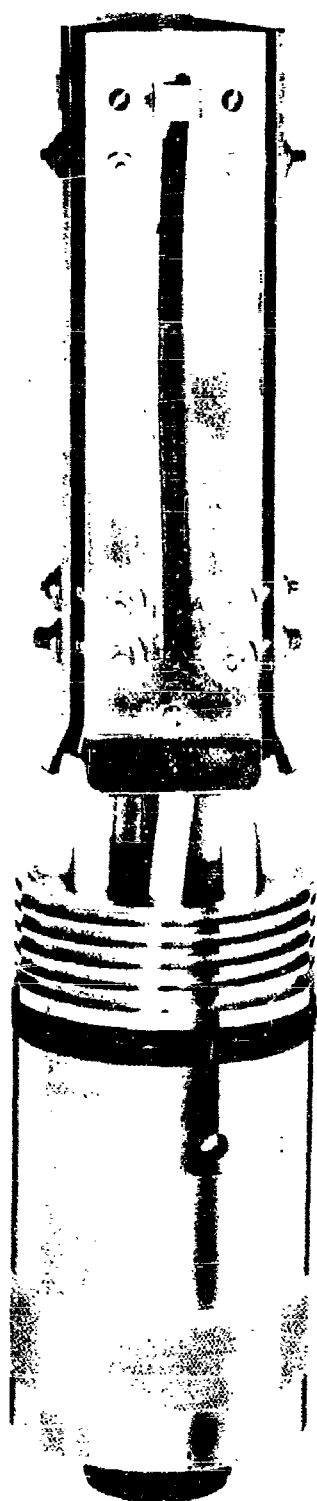


Figure 3. 3-Strand Test Cell Head.

required to reach acceptable mix homogeneity. Several of the mixers designed at ARC are shown in the following figures (Figures 4-7). The success of these mixers on a laboratory scale resulted in a decision to produce larger versions for semi-pilot plant batch sizes. These larger mixers have been and are being used at ARC and in many other installations.

In the intersecting cylinder mixers a high degree of safety and efficiency were combined, but this still did not satisfy the critical ARC users. Continued experiments with alternate designs were carried out until the cone-vertical concept was arrived at. It is with this design that we are particularly concerned today.

## II. DESIGN CRITERIA

### A. Symposium Recommendations

At about the time that the CV mixer was being developed a symposium at the U. S. Naval Propellant Plant of Indian Head, Maryland on "The Function of the Vertical Mixer in Solid Propellant Processing" was held. The conclusions of the Construction and Safety Committees of that Symposium were adopted as a guide in the design of the cone-vertical mixers. These conclusions are presented in Tables II and III. Except for the first item in the Safety Group's recommendations, these are still desirable goals today. Apparently, two schools of thought exist concerning vacuum deaeration. Therefore our design can be modified to accommodate either school's requirements. In the following discussion the design details of the CV mixers will be discussed and the means of satisfying the recommendations will be described.

### B. Description of CV Mixer Characteristics

The basic cone-vertical mixer concept is based upon a bowl constructed from a pair of intersecting  $30^\circ$  right circular cones whose axes are separated  $16^\circ$  as shown in Figure 8. Two conical helicoidal blades are used, the axes of which

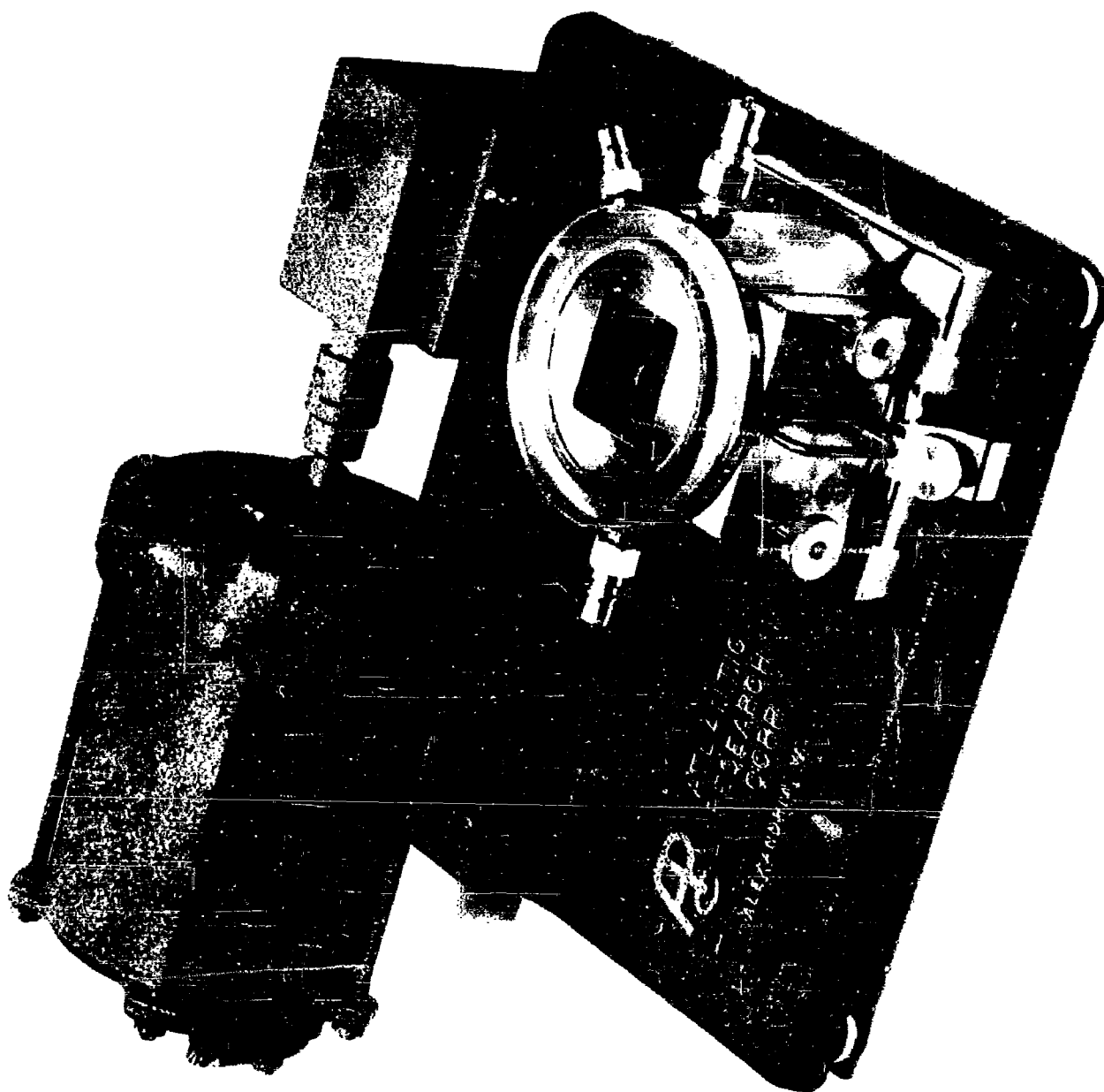


Figure 4. Model 4 LP Z-Blade Micro-Mixer.



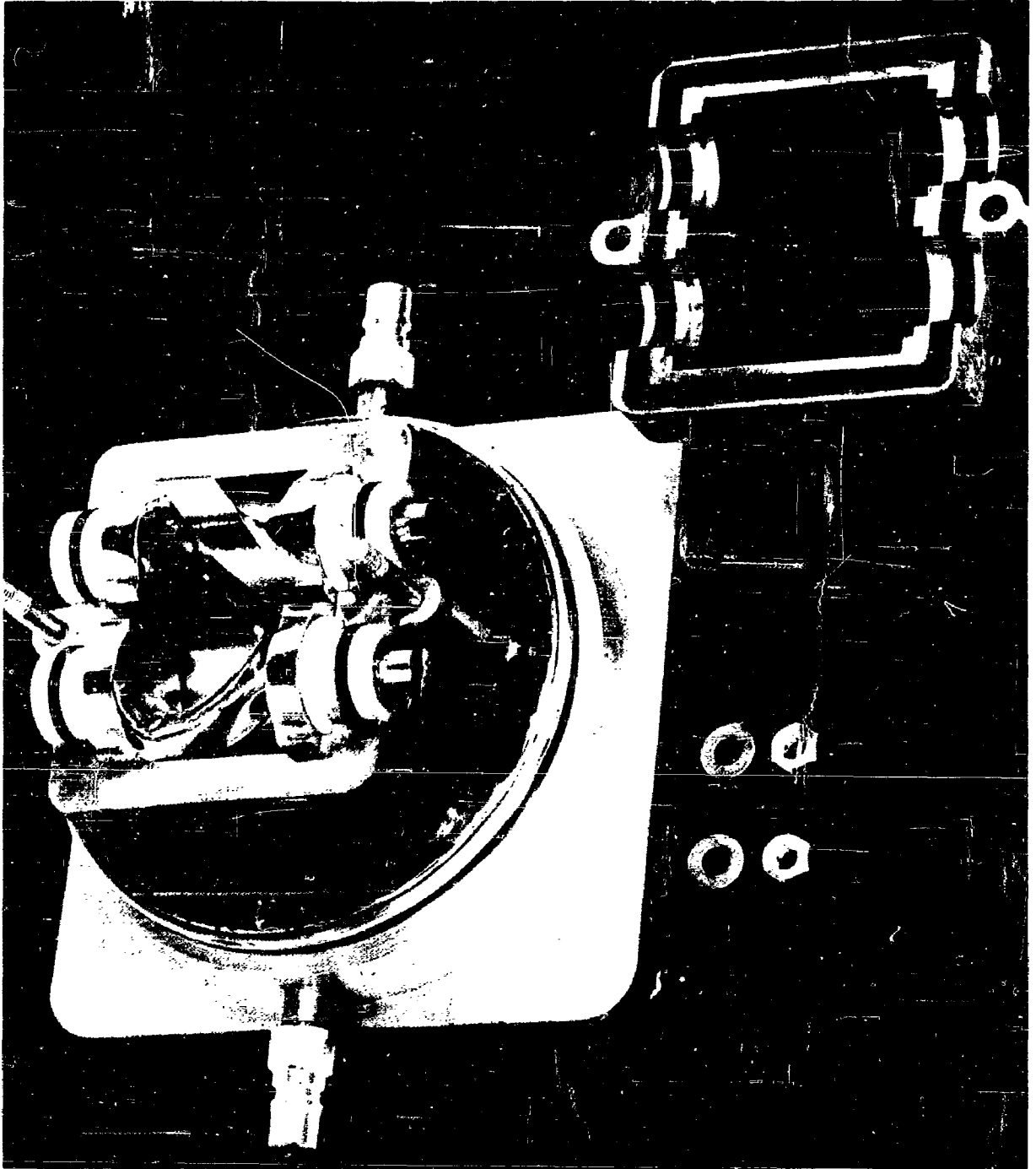


Figure 5a. Model 35 LP Z-Blade Laboratory Mixer  
(1 Pint Capacity) (Disassembled).

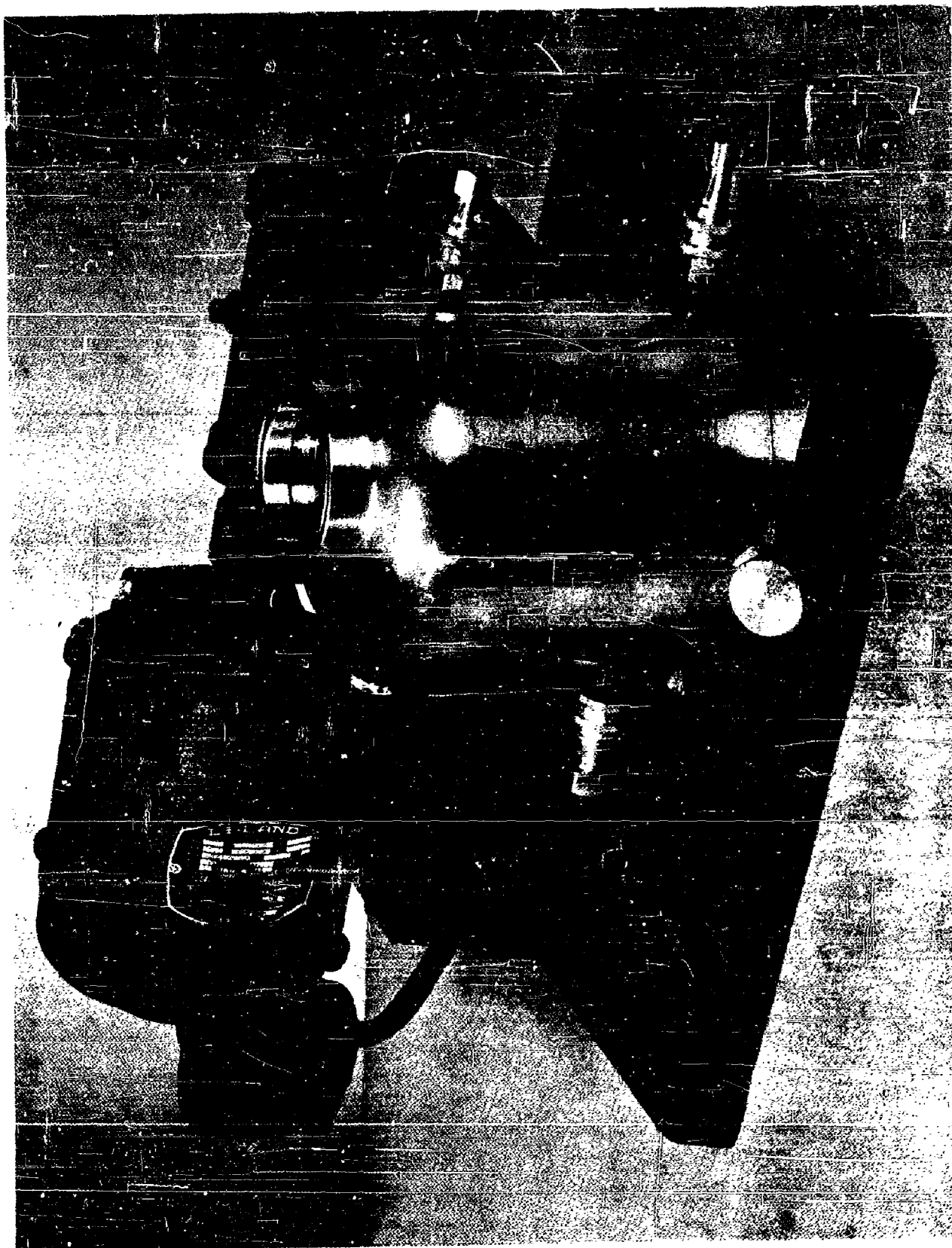


Figure 5b. Model 35 LP Z-Blade Laboratory Mixer  
(1 Pint Capacity) (Ready for Operation).

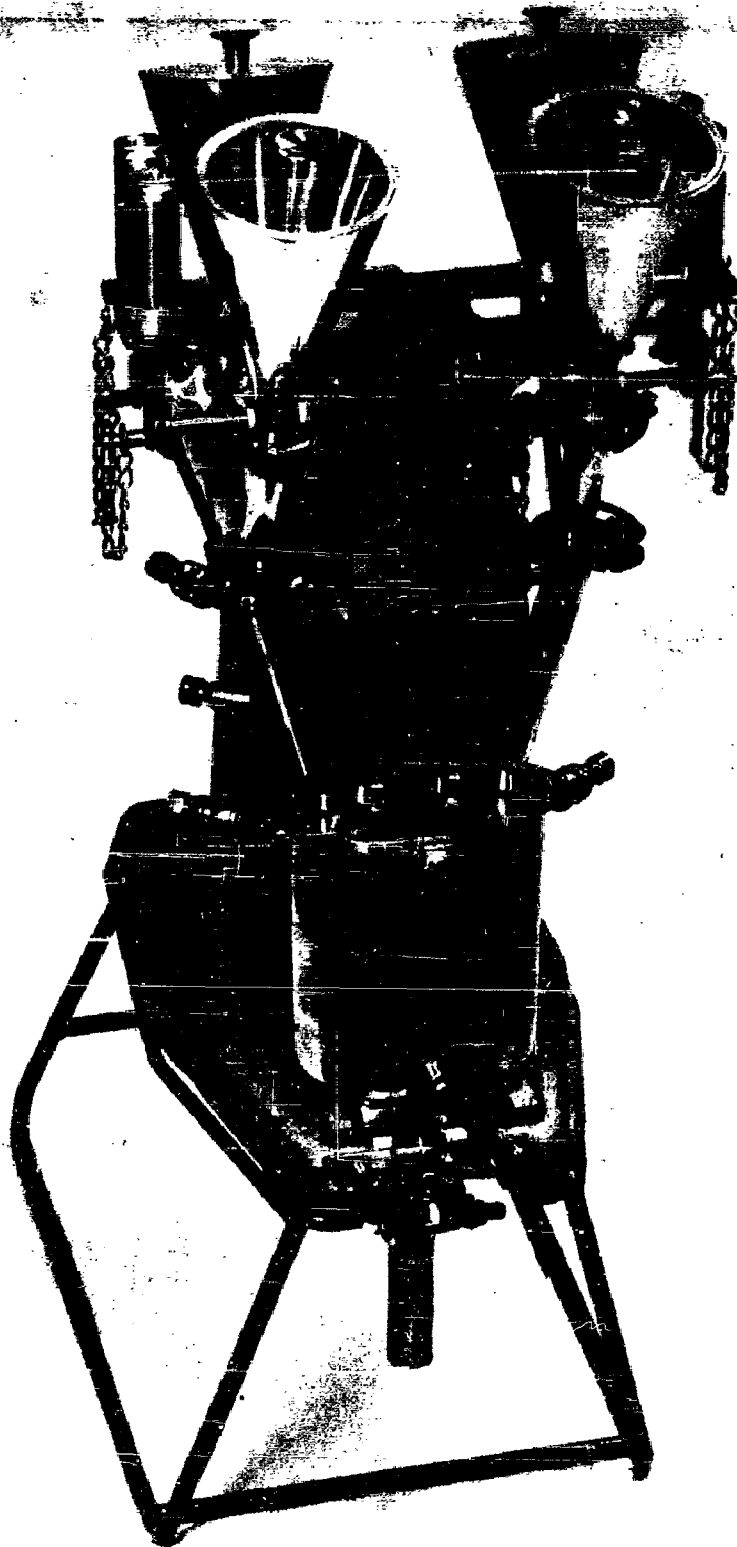


Figure 6. Model 60 LP Vertical Mixer for Remote Operation.



Figure 7. Model 1200 5-Gallon Vertical Mixer.

Table II. Construction Study Group Recommendations.

- STANDARD CONSTRUCTION
- 1/8 INCH MINIMUM CLEARANCE ON ROTATING PARTS
- DRIVE COMPONENTS ABOVE MIXER BOWL
- NUMBER 4 TYPE POLISH FINISH
- JACKETED BOWL
- NO LOOSE PARTS
- POSITIVE BOWL ALIGNMENT
- MAXIMUM NUMBER OF VENT PORTS
- BOTTOM DISCHARGE PORTS
- 0.5 PSIG MAXIMUM BLOWOUT PRESSURE

Table III. Safety Study Group Recommendations.

- AVOID VACUUM MIXING
- MAXIMUM VENTING
- COMPLETE ELECTRICAL GROUNDING
- ELIMINATE DUST FROM COMPONENTS
- RIGID INSPECTION OF COMPONENTS
- THERMOCOUPLE WITHIN BLADES
- ALL INTERIOR PARTS SMOOTH, HIGHLY POLISHED,  
AND WITHOUT CRACKS
- ADJUSTABLE BLADE SPEED
- MINIMUM BLADE CLEARANCE 1/8 INCH
- MAXIMUM BLADE DEFLECTION 1/2 INCH CLEARANCE

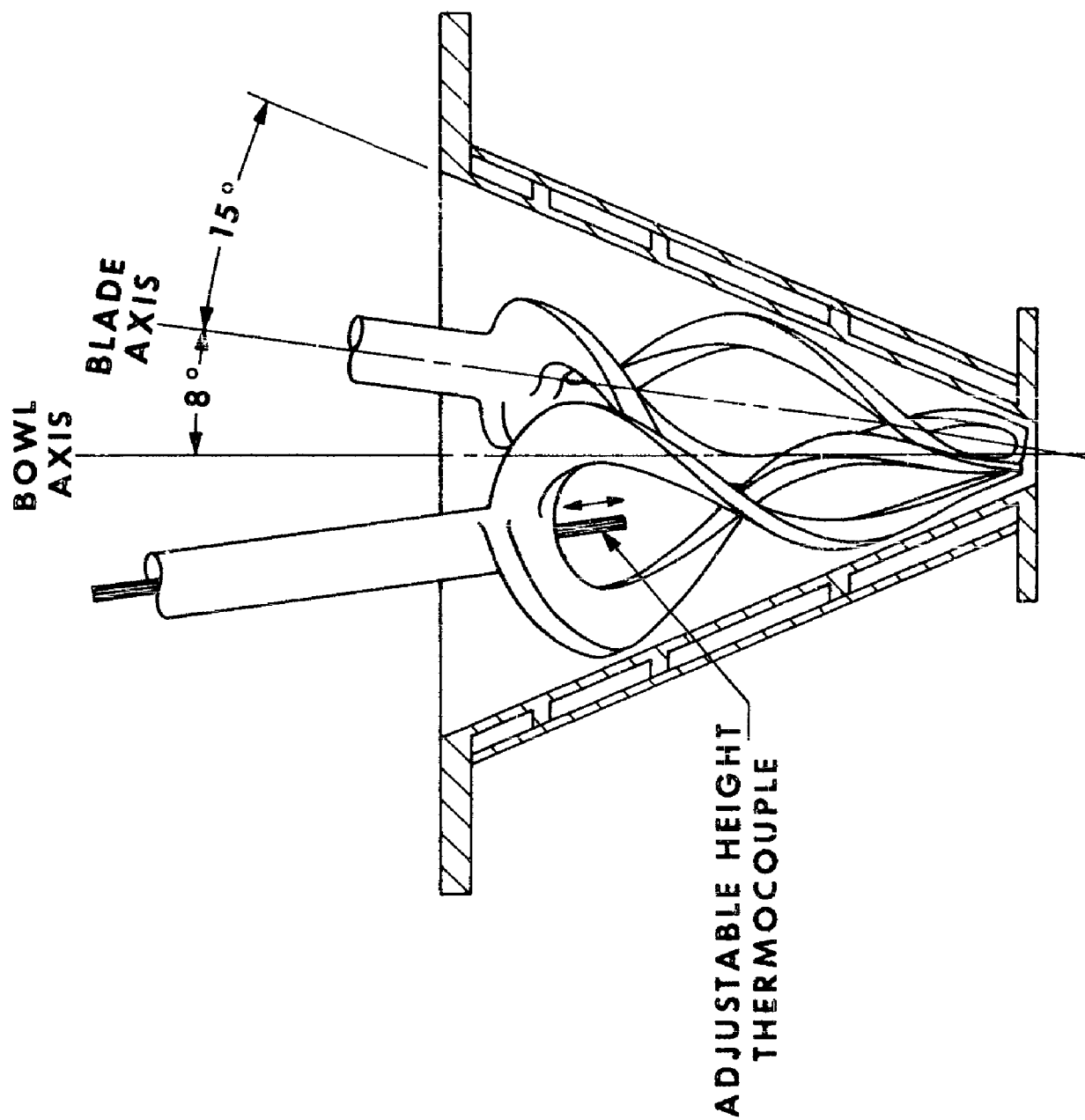


Figure 8. CV Bowl Construction.

coincide with the cone axes. With this basic construction several important safety features result. These are shown in Table IV, and Figures 9 and 10. In Figures 9 and 10 the ranges of adjustment of clearance as a function of blade movement are shown. Table V compares the ratio of exposed surface area to volume for the cone-vertical design with that ratio for other standard vertical mixers. A large value of this ratio is desirable since with a large exposed area deaeration can be achieved more rapidly with or without the aid of vacuum. In addition to these inherent safety features many others have been designed into the mixer and are made possible by the basic design. Some of these features are listed in Table VI.

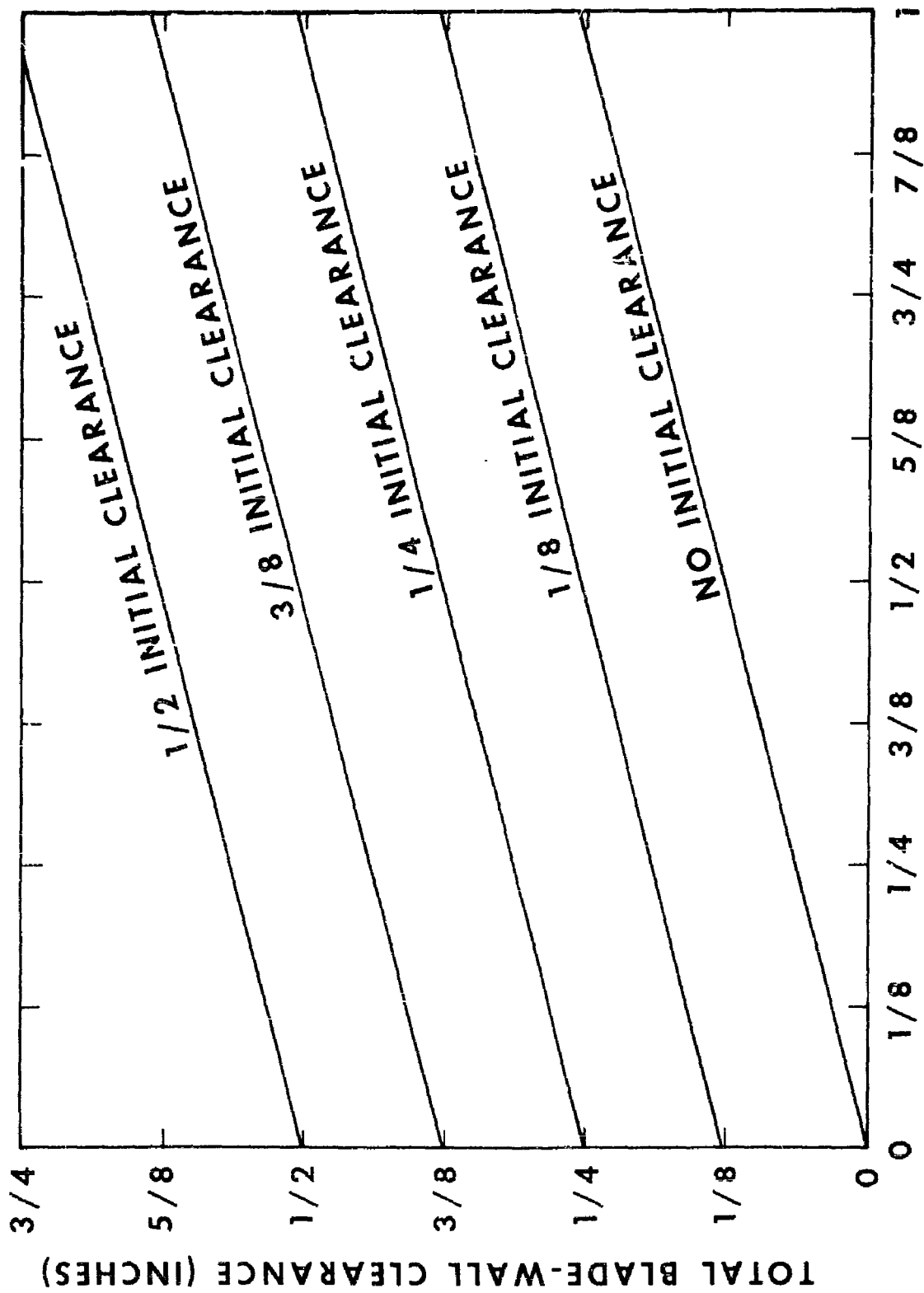
The first cone-vertical mixer was the laboratory model, different versions of which are shown in Figures 11 and 12. Specifications for this mixer are shown in Table VII. The mixing action for this and the larger mixers is shown schematically in Figure 13. Since the blades are basically screws of deep pitch, material is transported downward along the bowl wall and is then forced upward in the regions between the arms of the two blades. The counter-rotating interweaving action results in an extremely high dispersion rate with a low shear rate. Another feature of this blade design is the varying peripheral blade speed as a function of depth in the mix at constant shaft rotational speed. Curves showing this behavior are presented in Figure 14. These curves are for the 40-gallon mixer. As a result of the blade action and the peripheral speed change, extremely rapid mixing is achieved without the comminuting effect which is found in high shear mixers. This blade action is also of considerable importance in the discharge of material from the bowl. Natural extrusion is achieved due to this screw-like mixing pattern, when the bottom valve is opened.

Many of the important safety and other design features are best shown in Figure 15. The simple design and construction are readily apparent from this over-all view. Notice the compactness of the drive motor and the worm gear reducers. These components are commercial units that are readily available. The output shafts of the reducers incorporate dry well construction, thus obviating problems often encountered with other types of construction in which lubricants can drip into the mix. For ease of cleaning and to prevent unwanted



Table IV. Safety Features Inherent in the Cone-Vertical Design.

- **ADJUSTABLE BLADE-TO-BLADE CLEARANCE**
- **ADJUSTABLE BLADE-TO-WALL CLEARANCE**
- **LARGE RATIO OF EXPOSED SURFACE AREA TO VOLUME**
- **MAXIMUM COOLING WHERE MAXIMUM HEAT IS GENERATED**



AXIAL DISTANCE BLADES ARE RAISED TO  
OBTAIN ADDITIONAL CLEARANCE (INCHES)

Figure 9. Adjustment of Blade-Wall Clearance.

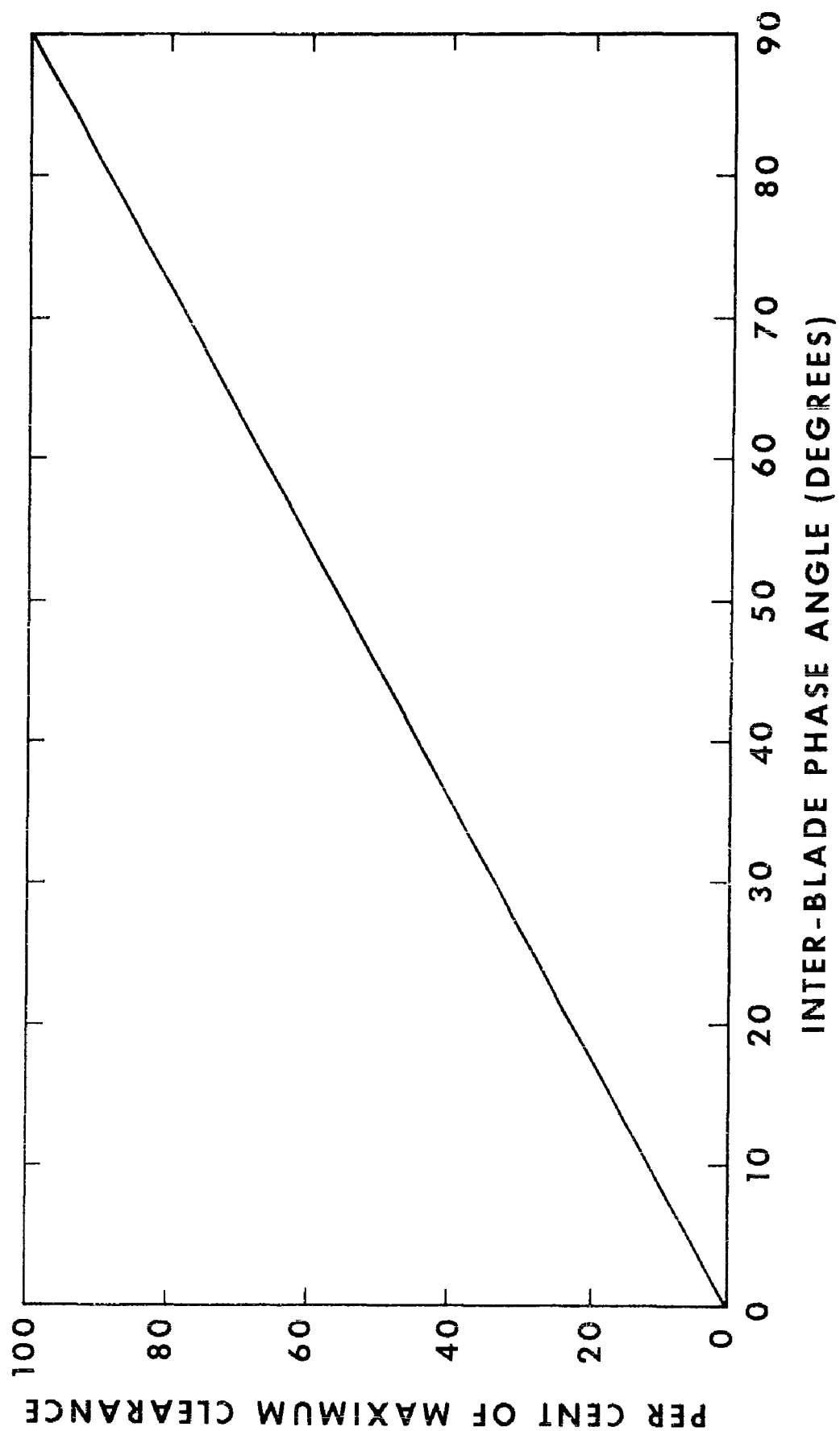


Figure 10. Inter-Blade Phase Angle Versus Inter-Blade Clearance.

Table V. Comparison of Area/Volume Ratios.

<u>MODEL</u>	<u>AREA / VOLUME RATIO</u>
<u>3-QUART MIXERS</u>	
ARC - 3 Qt	0.307
HOBART MODEL C-100	0.226
<u>3-GALLON MIXERS</u>	
ARC - 3 Gal	0.188
ROSS #130 ELs	0.133
<u>150-GALLON MIXERS</u>	
ARC - 150 Gal	0.0450
BAKER PERKINS	0.0220

Table VI. Safety Features Designed Into the Cone-Vertical Mixers.

- NO GEARS OR BEARINGS EXPOSED TO MIX ENVIRONMENT
- MINIMAL WEIGHT OF COMPONENTS ABOVE MIX CHAMBER
- SIMPLE DESIGN AND CONSTRUCTION
- EASY MAINTENANCE
- THERMOCOUPLE IN BLADE
- LIQUID ADDITIONS THROUGH BLADE SHAFT
- AUTOMATIC BOWL DROP IN CASE OF PRESSURE OR TEMPERATURE RISE
- REMOTE LOADING-AUTOMATIC HOPPERS
- REMOTE OPERATION
- REMOTE DUMPING AND CLEANING

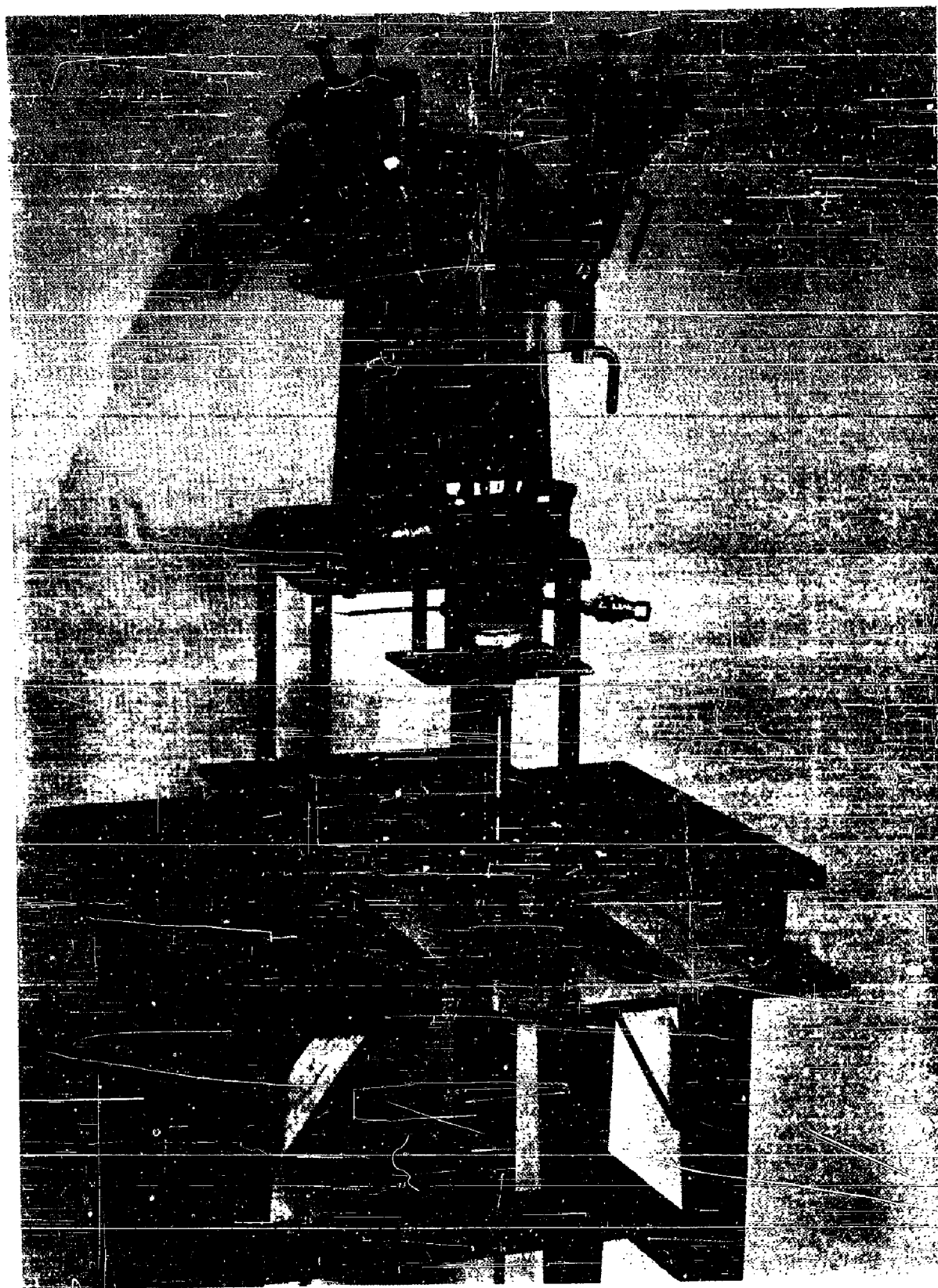


Figure 11. Model 2 CV Cone-Vertical Micro-Mixer.

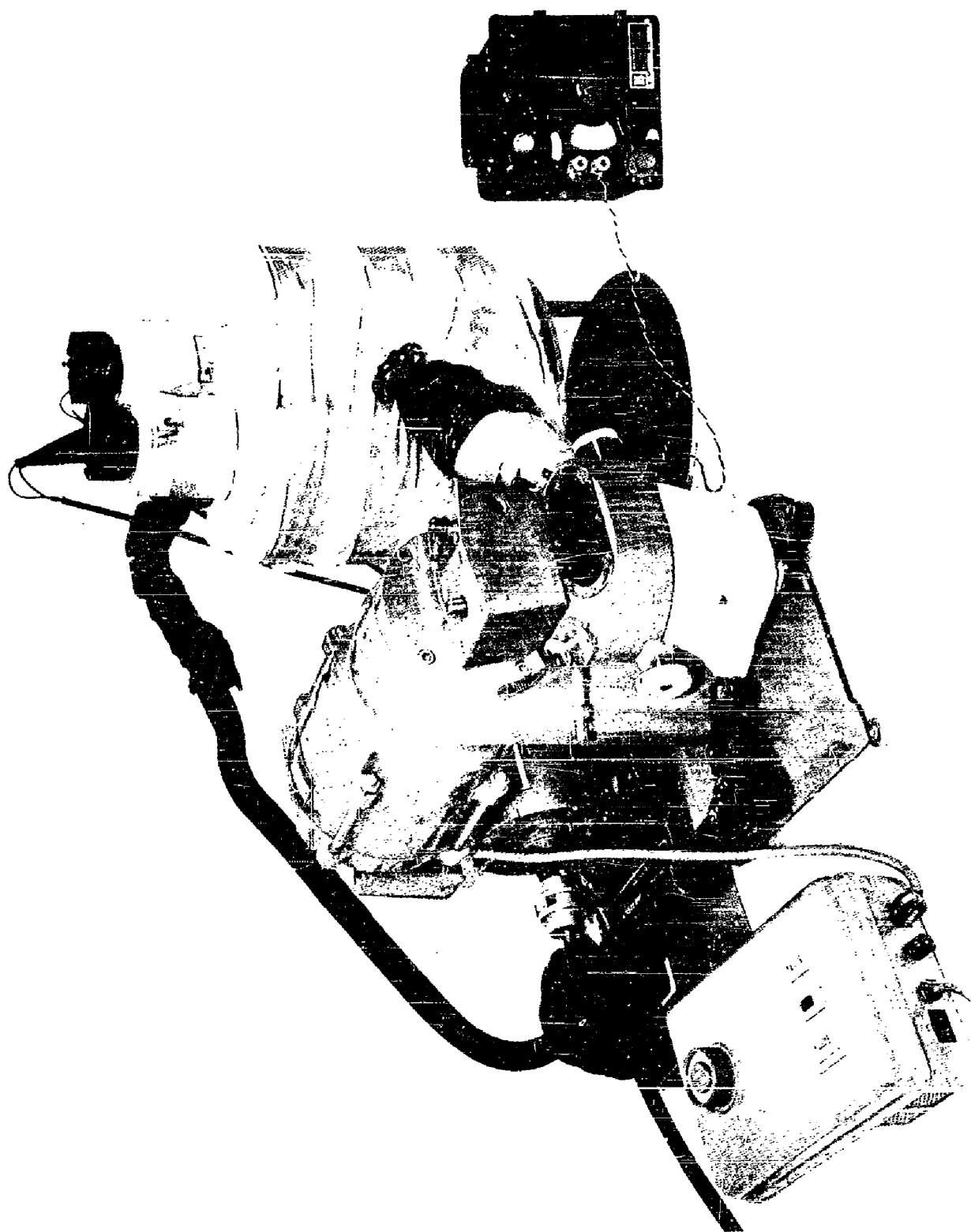


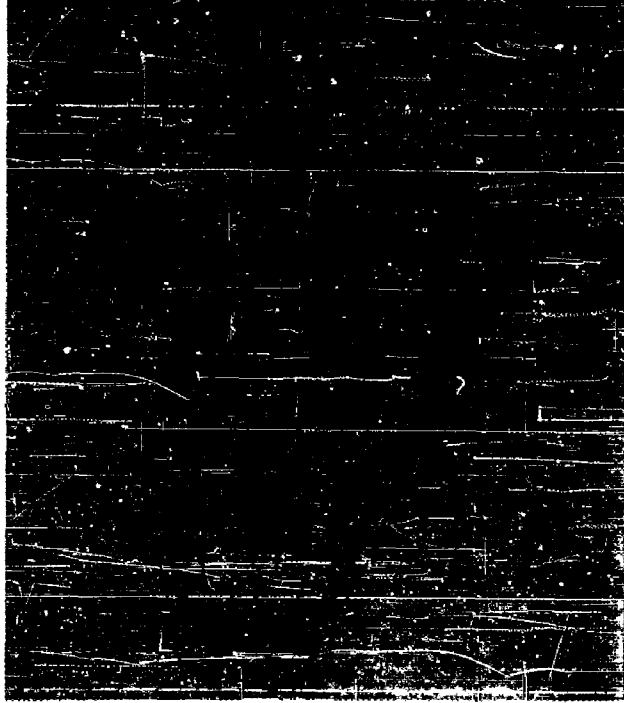
Figure 12. Model 2 CV Cone-Vertical Micro-Mixer for High Temperature Application.

Table VII.

# SPECIFICATIONS

TYPE OF MIXER		Vertical Micro-Mixer	
DIMENSIONS.			
Base	11 x 21 in	Height	23 in
COMPONENTS:			
Mixing Chamber		Drive Unit	
Total volume	370 cc	Gear box totally enclosed, water cooled	
Maximum mixing volume	150 cc	Slip clutch adjustable from 2-15 ft/lbs	
Minimum mixing volume	15 cc		
Operating vacuum	1-2 mm Hg	Motor	
Jacket pressure	150 psi	DC, 1/2 hp, 2,000 rpm	
Blades		Explosion-proof Class I, Group D	
Shaft speed	20-200 rpm	Class II, Groups F and G	
Tip speed	0.01-0.12 in/sec	Control Unit	
Clearance	0.062 in	115 volts, 50/60 cycles	
		Speed regulation 1-5% base speed	





**Figure 13. Mixing Action on CV Mixer.**

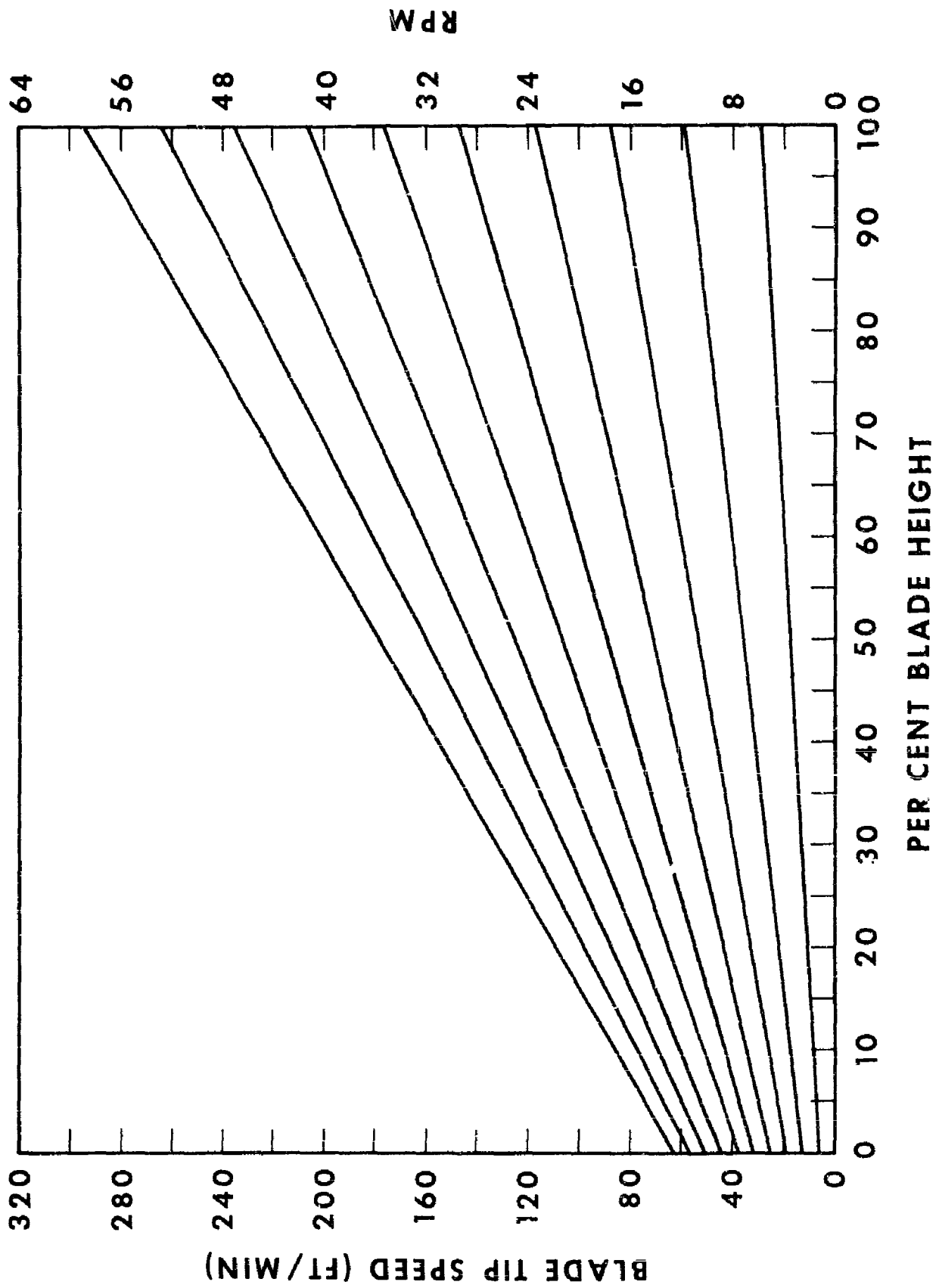


Figure 14. Blade Tip Speed Versus Blade Height (constant rpm).

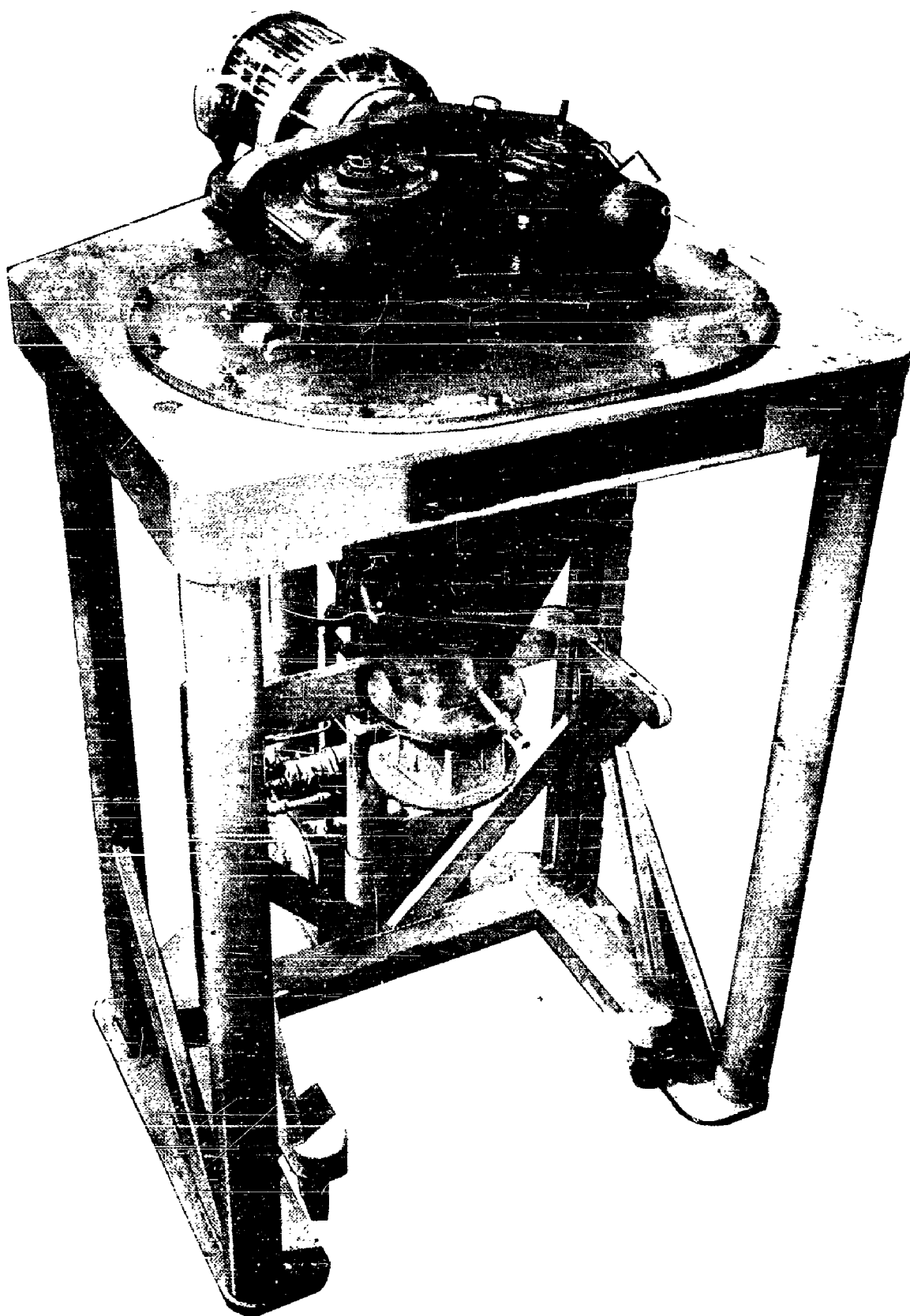


Figure 15. 40 CV Cone-Vertical Mixer.

corrosion problems all parts in contact with the mix are constructed of 300 series stainless steels, highly polished and pit free. Low carbon grades are used to permit simpler welding procedures and to avoid sensitization in heat affected areas adjacent to welds.

The relatively low weight of components above the head is of particular importance, and tends to reduce the danger in the event of an accident. Even less weight can be obtained, for those who are interested, by employing a fluid power motor to replace the electric motor drive. It is now possible to obtain such units operating in the range from 500 psi to 1500 psi with power output of the level required. Plans are being made to evaluate such units on the 8-quart mixer now being designed.

The two 6-1/2-inch diameter ports at the front of the head are designed for use with remotely operated hoppers for making additions to the bowl. Completely remote operation is contemplated. The 3rd port on top is designed to take a 3-inch vacuum line if vacuum deaeration is required. If a more open head is required a modified design will be available and is shown schematically in Figure 16.

All attachments on the head are made using welded studs thereby eliminating one common source of trouble. The reducer output shafts have been modified to provide 1/2-inch diameter axial holes completely through them. With this provision several important design features become possible. First the mixing elements can be attached to the speed reduction units outside of the mixer head by means of bolts which pass through the output shaft and thread into the blade shaft. This in turn permits variation of the depth of the blade in the bowl and therefore variation of blade-to-blade and blade-to-wall clearance by simply adding or removing shims from the bottom of the reducer shaft which is inside the bored out blade shaft as seen in Figure 17.

Figure 17 shows other features which are made possible by this design. For example, a thermocouple can be brought into the mix through the shaft or liquid additions can be made in a similar manner.

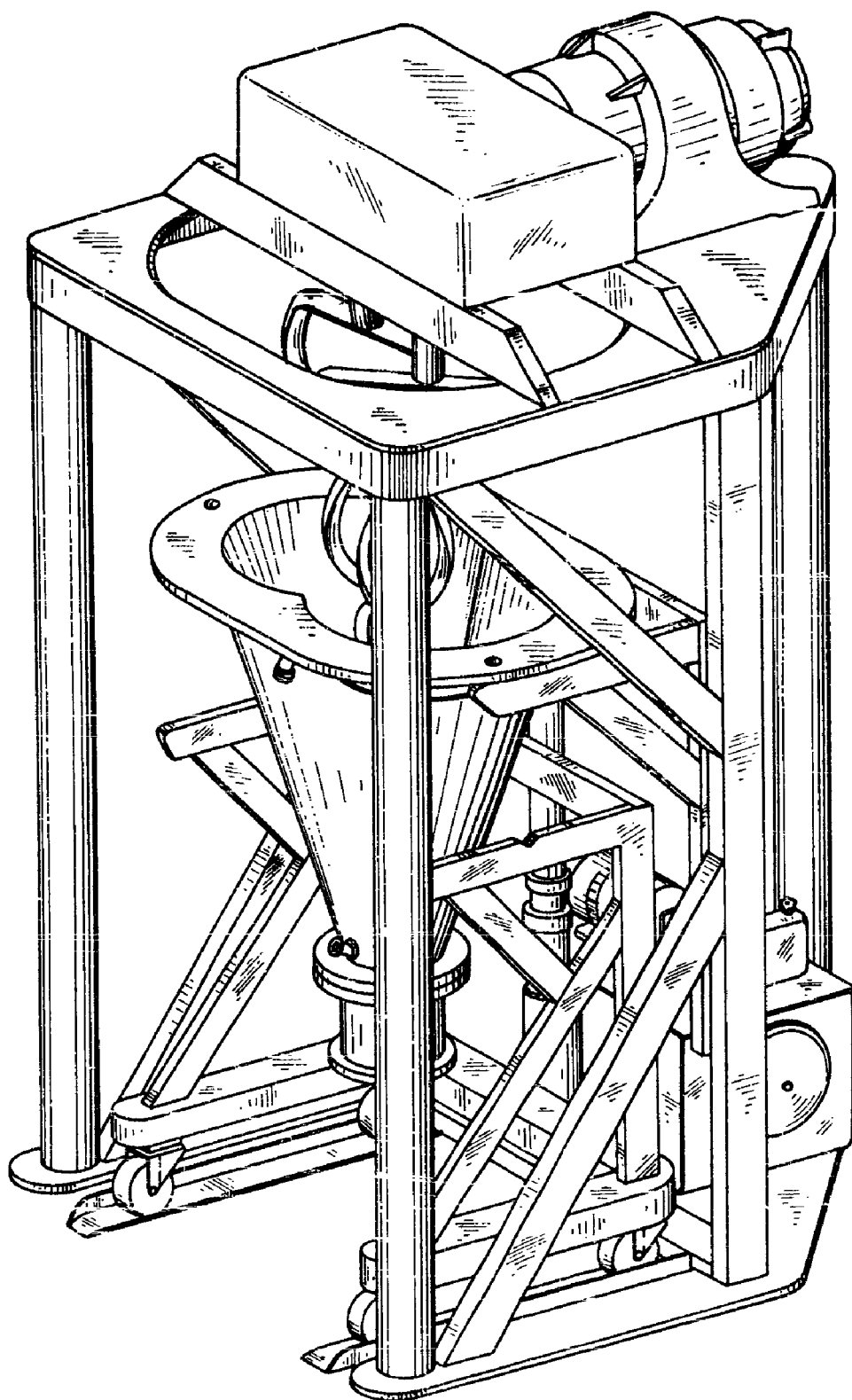


Figure 16. 40-Gallon Mixer Open Head Design.

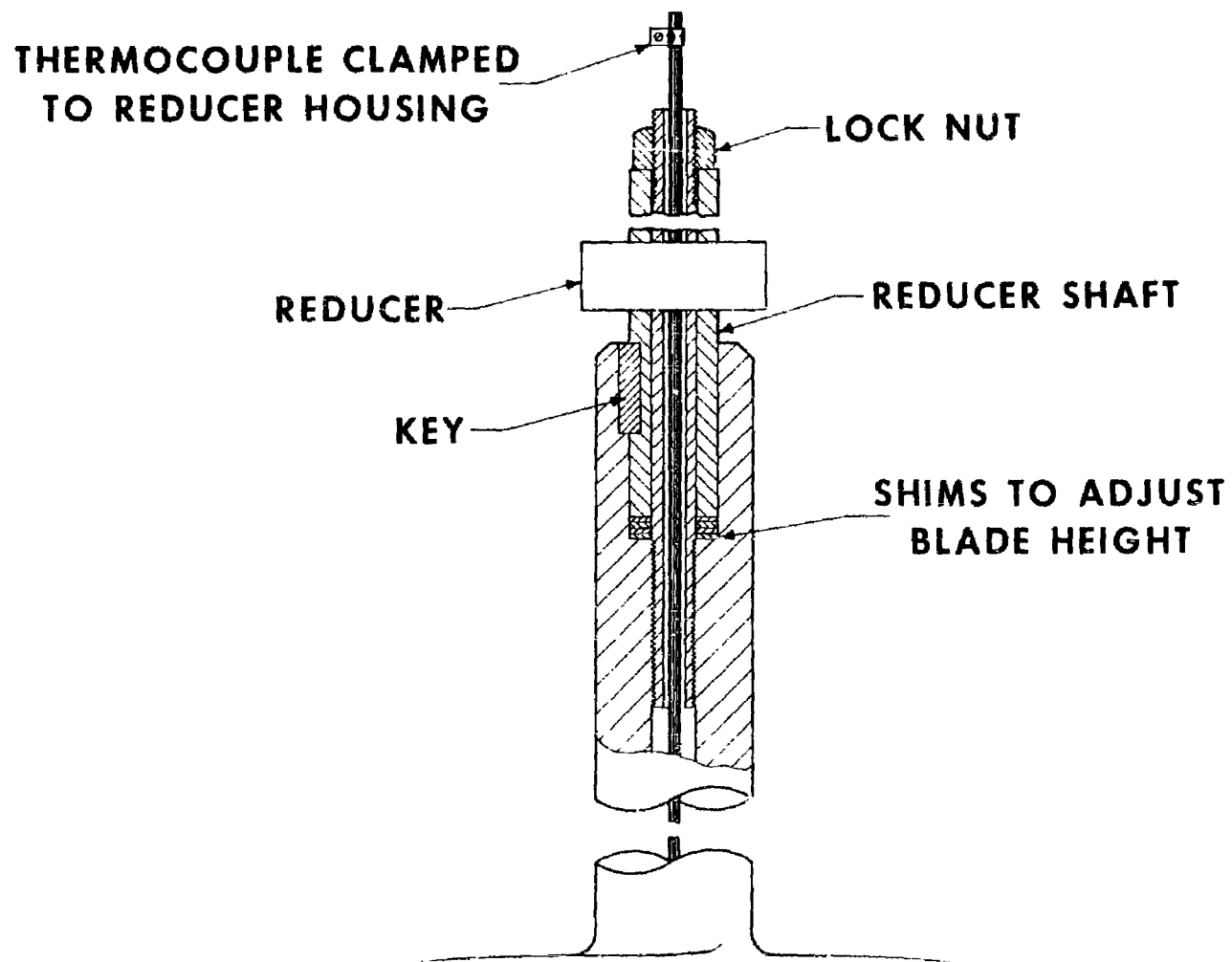


Figure 17. 40-Gallon Mixer Blade Shaft

The blades are of considerable interest from other standpoints also. They are cast of 304L stainless steel and have no structural welds. The castings are produced to ASTM Class I or II radiographic standards, thus assuring complete soundness.

To illustrate the extremely intensive mixing action of the blades we have taken a number of sequences showing the blades themselves and then looking up into the bowl from the bottom through a plexiglas window we can see their effect in actual mixing. For this experiment pure corn syrup was used. As the syrup is agitated, air is entrained as relatively large bubbles which are broken up into smaller and smaller sizes until their size and total number cause the previously transparent medium to become opaque.

Other features of this line of mixers are the method of raising the bowl and the positive bowl alignment holes in the bowl flange. The pins have been kept quite short in order to prevent hang-up of the bowl, a situation which can occur when long pins are used.

The bowl is fabricated completely of Type 304L stainless steel. It is jacketed to provide for either heating or cooling. Using 3/16-inch inner and outer walls 100 psi inter-wall pressure can be accommodated. A thermocouple is provided in the bowl to monitor mix temperature, but it is felt that this location is inferior to the blade-shaft location.

In the smaller mixers, until recently the bowls have been fabricated units, being machined from solid stock. Several bowls are now being prepared from castings, a procedure which should result in several improvements, safetywise, over the former fabrication methods, primarily in eliminating many welds.

Although they have not yet been installed on this unit, additional safety features will provide for interlocks between: (1) the cart and frame, (2) the bowl and head, and others. To prevent operation of the lift and blades unless the bowl is properly positioned, limit switches (explosion proof) will be positioned on the frame and tied in with the hydraulic power package and with the drive motor. With the bowl raised in its proper position other limit

switches will close circuits to the drive motor, the vacuum pump, and with the hopper valves (if they are provided).

Once mixing has started other interlocks between pressure and thermocouple gauges and drive motor, solenoid bowl drop valve and a deluge system will provide for stopping the blades, dropping the bowl and actuating deluge if pressure and/or temperature rises above preset limits. A remote control panel layout showing the incorporation of these features is shown in Figure 18.

Design work is now nearing completion on the 8-quart mixer which will be similar in design to the 40-gallon unit. It will incorporate all of the safety features that are common to the present units.

### III. EXPERIENCE

At this time 14 of the Model 2CV units are in use at various installations throughout the country. Table VIII is a list of users. The majority have been used in the processing of solid propellants or explosives without any known accidents caused by the mixer design. The potentials for this design are seen in the application of Goodyear, Koppers, and Battelle, where a variety of high viscosity polymers are being investigated. These laboratories are using units modified for high temperature, vacuum operations. At Goodyear, temperatures up to 500° F and vacuum of 0.1 mm Hg abs are maintained while processing materials having viscosities in the range of 10,000 to 20,000 poise. In tests at Koppers this mixer has handled materials with viscosities between 20,000 and 40,000 poise. The larger mixers have been designed to provide the same capability.

The major use at ARC has been in mixing slurry fuels with high solids contents. In these systems proper mixing is related to viscosity with minimum viscosity being sought. Until the 2CV unit was made available a Hobart unit and an ARC intersecting cylinder mixer were used. To reach the proper viscosity with the Hobart at least 10 hours mixing time was required. In the 60 LP the mixing time for minimum viscosity was reduced



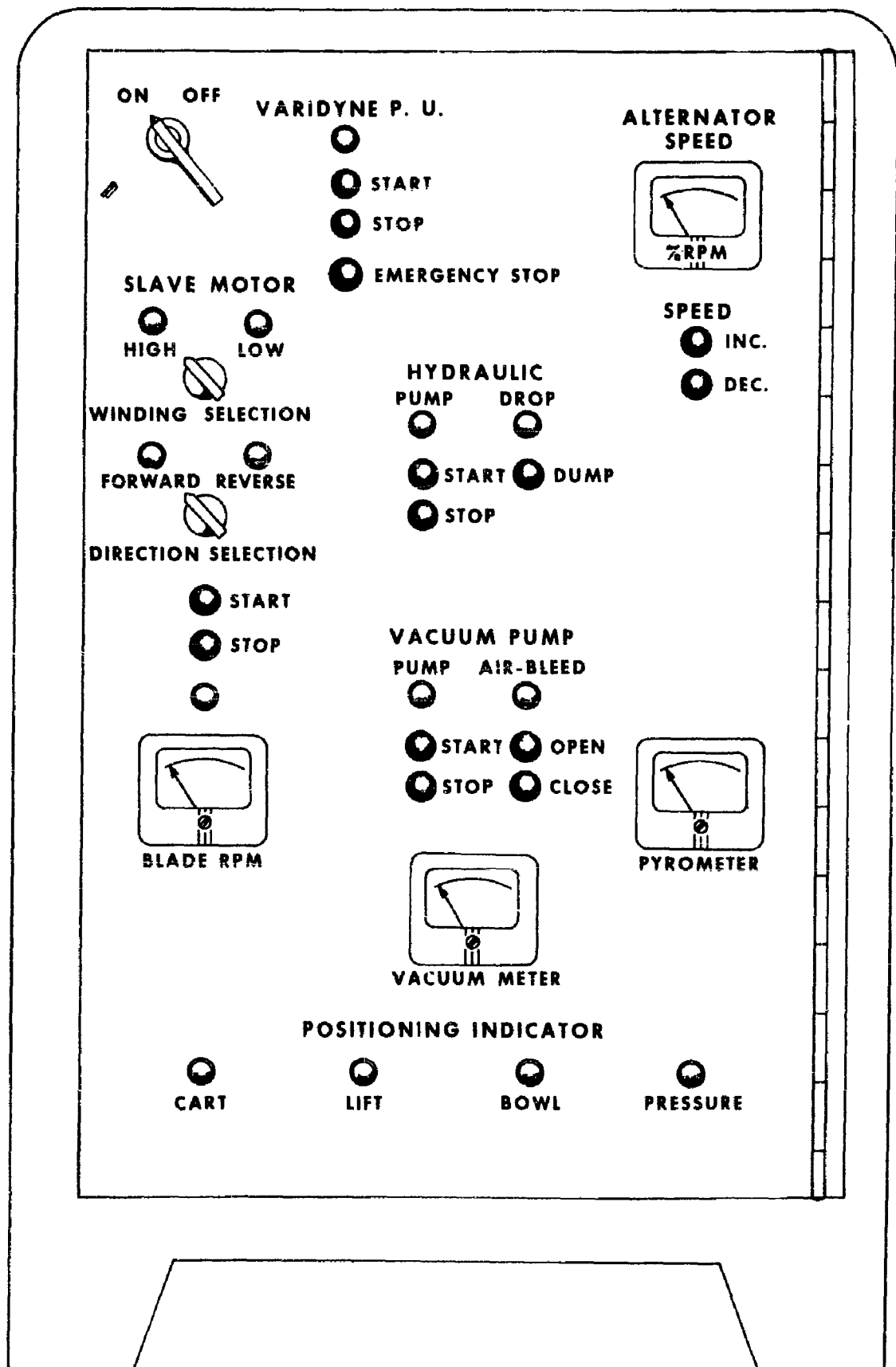


Figure 18. 40-Gallon Mixer Remote Control Panel.

Table VIII. Users of the Model 2CV Micromixer.

- ARC - PINE RIDGE
- AMERICAN CYANAMID
- UNITED TECHNOLOGY
- ARC - NUCLEAR ENGINEERING DIVISION
- NAVAL PROPELLANT PLANT
- EDWARDS AIR FORCE BASE
- MARQUARDT
- GOODYEAR (HT)
- KOPPERS (HT)
- BATTELLE (HT)
- ARMY MISSILE SUPPORT COMMAND
- HERCULES POWDER-ALLEGHANY BALLISTICS LABORATORY
- NASA/LANGLEY
- AEROJET GENERAL

to 5 hours. With the 2CV a further reduction to 2-1/2 hours resulted and in addition lower viscosity was obtained.

Recently several of these slurry mixes were made in the 40-gallon unit. In jumping from the 150 cc mixer to the 40-gallon unit, the volume scale-up was approximately 1000 to 1. In the small unit, optimum results were obtained at 130 rpm with about 70 per cent of the bowl volume filled. The equivalent blade speed in the large mixer based on peripheral speed was 30 rpm. The resulting mix viscosities in 2-1/2 to 3 hours, were even lower than had been obtained in the small unit despite much greater blade-to-wall and blade-to-blade clearances.

These results prompted a recent investigation of the effect of blade clearance on mixing efficiency in the 2CV mixer. In this unit normal clearance is 1/16-inch. With clearance increased up to 1/4-inch preliminary results indicate that equivalent viscosities are obtained in the same mixing time. These tests are continuing, initially with slurry mixes, but eventually both inert and live propellant mixes will be studied.

#### IV. FUTURE

Although the blade configuration presently in use on the 40-gallon mixer appears to be extremely efficient, the design is a completely arbitrary one based only on experience with the 2CV mixer. In order to establish a firmer basis for optimum blade configurations for different types of mixing operations, an experimental program has been planned and will be instituted soon. In this program, the primary considerations will be related to safety, but mixing efficiency and mixing mechanics will receive substantial attention. These are the primary considerations that will determine the optimum blade configuration in the mixing of sensitive materials.

While it is recognized that these factors are interrelated, it will be more convenient to discuss them as separate and separable entities. In the sections below they are discussed on this basis.

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## Safety

Since it is imperative that metal-to-metal contact be avoided during the mixing of sensitive materials, it will be necessary to determine the conditions and the geometries which when combined will result in safe mixing. For any given blade configuration there are three variables that will determine the tendency towards blade-to-blade or blade-to-wall contact. These are:

- a. The initial clearances,
- b. The forces acting on the blades, and
- c. The ability of the blades to resist these forces.

In the cone-vertical mixers both blade-to-blade and blade-to-wall clearances can be adjusted. Since these clearances will be important in determining mixing efficiency and mixing mechanics also, it is planned that a wide range of clearances be investigated.

The forces acting on the blades will be determined by the blade speed, both rotational and peripheral, the viscosity of the mix, and its weight. These forces will result in torsional stresses, both in the shaft and in the helix. Those in the helix will tend to twist it further, resulting in axial compression of the blade. Any imbalance of forces acting on the individual arms of a blade will tend to cause axial deflections. In the experiments planned, these factors will be considered and attempts will be made to determine the effects of speed, viscosity, loading and imbalance on the change in clearance during mixing.

Several methods of determining blade-to-blade and blade-to-wall contact during mixing have been suggested. Among these are: (a) the measurement of resistance decrease in an open electrical circuit between the blades, (b) the use of direct reading temperature indicating stickers, (c) the use of thermocouple probes, and (d) the use of coatings which would show areas of contact. Of these (b), (c) and (d) show most promise. The

Pyrodyne "Temp-Plates" contain heat-sensitive elements that are hermetically sealed in laminated, high-temperature-resistant plastic which are also resistant to solvents, fuels, grease, oil, water, and steam. "Temp-Plates" are available in the temperature range from 100° to 500° F in 10° F intervals. If the indicator reaches its stated temperature its color changes from pastel to black irreversibly. Since rubbing contact between two solids usually results in a large local temperature rise, such indicators may permit detection of even light contact between blades or between blade and bowl. Photoelastic or pressure sensitive coatings may provide useful information in a similar manner.

A combination of thermocouple probes and such indicators as described above are now planned for monitoring both temperature and contact. Thermocouples will be mounted on the bowl inner wall, while indicators will be mounted on the bowl wall and on the blade in locations most likely to contact.

The blade configuration required for resistance to deformation will be based upon provisions for stiffening with respect to axial extension or contraction and for strengthening with respect to torsional loads exerted. The use of a central shaft for providing axial stiffening will be investigated.

### Mixing Efficiency

Mixing efficiency can be defined on the basis of a comparison of the degree of dispersion actually obtained with perfect dispersion. Since the perfect dispersion is unattainable and from an economic standpoint undesirable, only qualitative judgment or determinations of the mixing efficiency are possible. However, by comparing the time required to reach a desired degree of dispersion or a desired consistency under different mixing conditions, a measure of efficiency can be obtained.

Since it is necessary that all material within the container undergo sufficient movement to accomplish the desired degree of dispersion, the factors that will affect this movement are those that must be considered. These factors include the viscosity of the mix, the rate of movement of the mix, and the amount

of shear imposed upon it. Mix viscosities will be determined by the relative amount of solids and liquids and interactions between them. The rate of movement of materials will be determined by the rotational speed of the blades and by the lead angle of the helix, while the shear will be determined by the clearance between blades and between blade and bowl as well as by the rate of movement of the mix relative to blades and bowl.

There are certain limitations imposed by fabrication considerations and by past experience on the spectrum of configurations that will be covered. Therefore it is planned that a constant blade pitch be maintained and that the twist be between  $180^\circ$  and  $360^\circ$ . In order to move as much material as possible per revolution of the blade, the frontal area of the blade in contact with the mix should be maximized for any given cross-sectional area of blade. Fortunately, torque requirements also favor this configuration. Therefore, rectangular or trapezoidal sections with width greater than thickness will be the shapes investigated.

### Mixing Mechanics

"Mixing Mechanics" is a contrived term used to denote certain phenomena incidental to the mixing operation, but intimately associated with it. Most important of these are the temperature rise in a mix and the pressures developed during mixing. The heating effects are the result of the work performed by the blades in the movement of material, in decreasing the entropy of the system, and in friction. If exothermic reactions are involved, these two contribute to heating effects.

The other so-called mechanical effect is the pressure build-up in the mass of material. This is felt to be an important consideration in the cone-vertical mixer design since it acts in many respects as an extrusion apparatus.

In order to establish the extent of pressure build-up in the mixing process, pressure transducers will be installed in strategic locations in the mixer in order to monitor the pressure history as a function of the several variables being investigated.

## Analysis

An important part of this program will involve an analytical phase. The blade designs will be subjected to a thorough stress analysis to ensure that adequate strength and stiffness are achieved for any anticipated condition. The results of the stress analysis for the various blade configurations will be compared with deflections measured during actual mixing as well as in static tests as a check on the validity of the analysis for future design conformation.

A second analytical study will involve the factors involved in the scale-up of the mixer from laboratory scale to intermediate and larger prototype models. This study will involve those parameters peculiar to mixers and also those associated with extrusion since the CV design is a combination of the two.

## Larger and Continuous Mixers

Also planned for the future are an extension of the CV mixer line to include production mixers in the 150-gallon and 300-gallon size range. Beyond that the ultimate aim is to take advantage of an additional feature inherent in the basic design. This is its ideal configuration for continuous flow and of course the goal is continuous mixing.



Mr. Colitti: I wonder what kind of deflections they have experienced with the viscosity mixes up to 40,000 poise?

Mr. Arbiter: In this model our initial clearance is about 0.063 and we haven't had any indication of any measurable deflections. In the figure of application, any impurities that you might get into the mixer would show up as a discoloration and if there are any deflections which would cause the blade to contact the wall of the mixer they would give discoloration. Apparently there haven't been any specific measurements made of the deflection on this particular mixer started with this small deflection. Apparently there isn't any large deflection.

Mr. Colitti: How about your 40 gallon mixer?

Mr. Arbiter: 40 gallon mixer we have. We do get some indication of deflection which was due more to the installation. We had to shim up our reducer on two blades. In one case the blade was tied down and there was no deflection at all.

F I L M  
OPERATIONAL SHIELDS TESTING  
BY  
CHARLES R. GOFF  
LONE STAR DIVISION  
DAY & ZIMMERMANN, INC.

The motion picture film titled "Operational Shields Testing" was put together originally from various film strips of tests conducted at the Lone Star Army Ammunition Plant, operated by Day & Zimmermann, Inc., for informational purposes of supervisors who might use such shields, yet not be in a position to view such testing. When this film was first assembled, it was decided to add sound in order to provide a more realistic duplication of the actual testing of such shields.

Tests of the adequacy of operational shields have always been a part of Lone Star's Safety Program, but the use of high speed motion picture cameras has only been utilized for the past year and a half. These cameras operate at approximately 2,500 frames per second thereby affording qualified management personnel exact evidence of the results of each test.

As is shown in the film, the personnel shelter is constructed of 12 inches of reinforced concrete and adequately protects all personnel during such testing. Prior to the use of high speed motion picture cameras, craft paper would either be positioned on wood supports around the operational shield being tested, or fastened to the outside of the door, or in some cases, the door area would be painted white as a means of determining, after the test, whether or not any leakage occurred. In other cases the operational shield being tested was positioned a sufficient distance away from the personnel shelter to preclude the possibility of shrapnel reaching the shelter and qualified observers with binoculars would observe the test from within the personnel shelter and through plexi-glass protected apertures to determine if the operational shield was adequate.

This procedure seemed adequate until our first use of the high speed motion picture cameras. After this first test was made, the qualified observers stated that the operational shield held and was acceptable for installation. After the film was developed and viewed, it was noted that the door opening leaked flame requiring substantial changes to the horizontal sliding type door required in this particular operational shield. Since that time, every operational shield test conducted at Lone Star has been filmed with high speed motion picture cameras.

It is recognized by management personnel of Day & Zimmermann, Inc., that numerous tests of operational shields have been made in the past by all Plants in order to develop adequate operational shields. In view of this, the necessity of our tests has been questioned and considered as duplication of

effort. Lone Star management personnel does not agree with this concept. For one thing, basic explosives are becoming more powerful. A good example is the comparison between dextrinated Lead Azide and RD-1333 Lead Azide. One piece of 1/2 inch plexi-glass will contain a given amount of dextrinated Lead Azide placed a certain distance away from the plexi-glass. The same amount of RD-1333 Lead Azide placed in the same position will shatter the same thickness of plexi-glass. RD-1333 Lead Azide has three times the penetrating power of dextrinated Lead Azide from a detonating velocity standpoint. In addition, when an operational shield requires, for example, a sliding type door, the depth of the slide-well and the amount of overlap of the door in relationship to the opening in the operational shield proper, is a critical factor and definitely has been proven by our tests to be the difference between an operational shield that entirely contains all fragments and flame as against an operational shield that does not contain all fragments and flame.

Furthermore, and as an example, tests made at Plant "A" show that the design of an operational shield is entirely adequate for 2 ounces of a given explosive. Assuming that information is available to management personnel and their needs were for 4 ounces of the same explosives, there is no accurate formula that will insure adequate operational shield protection for 4 ounces of explosives based on test results for 2 ounces of the same explosives. This is one more reason we intend to continue such testing of operational shields for all suspect explosives we process.

The use of such high speed motion picture cameras at 2,500 frames per second can result in a considerable savings in money and time as well as insuring safety of operating personnel by pin pointing for the design engineers, the exact point at which either the leak appeared or the fracture of the operational shield occurred. This is determined at the time the films are reviewed and at the moment of explosion, the camera is stopped and the film turned frame by frame by hand. If the initial fracture can be pin pointed, in many instances an angle iron file, for example, can be welded in place thereby adequately preventing the initial rupture. If, as was the case in the past, management personnel had to review the remains of the operational shield tested, much assuming was done as to corrective measures necessary and in many cases much larger and heavier and thereby more expensive shields were constructed than was necessary. This was also true when craft paper was used to surround the shield as in some cases the paper was ripped and it was extremely difficult to tell whether this was caused by pressure escape or shrapnel. High speed motion pictures can in many cases, as is shown by the film, track the shrapnel as it escapes from the operational shield.

In this film, we have reversed two different tests and the narrator alerts the viewer when these reversed film are to be shown. Please note

that in the first view of this test, it is difficult for the eye to tell exactly the first point of leakage even at 2,500 frames per second. Compare that part of the film with the one immediately following, showing the same test in reverse and you will see that your eye can then follow the first escape of flame exactly to the originating point. Even high speed motion picture cameras at 2,500 frames per second cannot in all cases slow the action of a high order detonation to the point where a person's eyes can adequately follow movement of flame or shrapnel.

In the film, tests are made using both the M1A1 electrical squib and special electric blasting caps as firing mechanisms. The reason for this is that in the testing of Propellants M-5, M-9, and M-10, normally you should only expect a flash and this can be better simulated by using the M1A1 electrical squib. We have experienced high order detonations using the M1A1 electrical squib in our tests, therefore, in order to create for test purposes the maximum hazard condition possible, we not only put a 25 percent overage of the explosive being tested in the shield but also utilize the special electric blasting cap to cause high order detonation if that is possible. In that way, if after installation, a condition is created in the operational shield which could cause high order detonation, then we will have already proved by tests such as these that the shield is adequate for the most severe condition possible.

The Lone Star Army Ammunition Plant, operated by Day & Zimmermann, Inc., considers such tests a normal part of the pre-planning required prior to an item being produced. The management group responsible for the review of such film consists of the Plant Manager; the Director of Safety & Plant Protection; the Director of Production; and the Production Manager where this equipment is to be used; the Director of Technical, Quality Control, and Value Analysis; the Director of Engineering and Maintenance; and the Contracting Officer's Representative Safety Director. This is the management group that will make the decision as to whether or not the operational shield is adequate for installation.

The tests on operational shields are conducted in accordance with a written Technical Program describing in detail the operational shield being tested, type and amount of explosives the shield is being tested for, and exactly how the test is to be conducted including personnel and explosive limits. These Technical Programs must be approved by the Technical Director, Safety Director, the Plant Manager, and the Contracting Officer's Representative. The Technical Program as written, effectively controls how each test is conducted and gives it official status. When tests are completed, the results of such tests are made a part of the Technical Program insuring a permanent and comprehensive report for future information. In addition, copies of such results are forwarded to Safety, APSA for their information and record.

The high speed motion picture camera used to make the film is the Fastax Camera, Model WF-3, manufactured by the Wollensak Manufacturing Company. This is for informational purposes only and not as a recommendation of the particular manufacture as other high speed motion picture cameras are available which would be equally efficient.

Before each test, as shown in the film, information as to the kind of explosive, weight of powder, shield dimension, shield material, firing mechanism, and test setup information is shown. Immediately after the film of the test is shown, information as to the results of the tests and the conclusions are also shown. Such tests of operational shields are repeated for each engineering design change required until the operational shield being tested completely contains the entire explosive charge.

In this film you will note two different designs of funnels for containing this Propellant. The conical shaped funnel has been tested and found to reduce the possibility of high order detonation in comparison with the tall cylindrical type funnel. Tests have not yet been completely evaluated on the final design of such hoppers and it must be assumed at this time that the reason for the reduction in probability of high order detonation, is the result of the lesser column height of a given weight of powder immediately over the actual point of ignition. By reducing this column height, the flame can then travel at a more horizontal level with the subsequent reduction in confinement and also heat build-up, thereby permitting a portion of the Propellant Powder to be blown out of the hopper unburned. Tests of the conical shaped hopper to date have shown a considerable portion of unburned powder granules within the operational shield whereas tests of the cylindrical type hopper do not have any unburned powder granules after the test.

The Lone Star Army Ammunition Plant uses a considerable amount of initiating type explosives in the items produced and although this film only shows tests of operational shields for Propellant Powders, similar tests have been conducted on initiating type explosives. Since it must be expected that in the loading of items with initiating type explosives, high order detonations will occur in the normal course of such loading, adequate operational shields must be utilized in producing such items. The frequency and severity of such explosions with initiating type explosives requires adequately tested operational shields in order to prevent to as great an extent as possible injury to operating personnel.

As is also stated in the film, because of time, this film only shows a small portion of such tests. The Lone Star Army Ammunition Plant operated by Day & Zimmermann, Inc. has developed many entirely automatic and remote control operations involving the handling of hazardous explosives,

but much work still needs to be done and may never be entirely complete as regards the complete mechanization for the loading of such items because of insufficient lead time and other limiting factors. In the meantime, we will continue to view each explosive used, with suspicion, and attempt to adequately protect the operating personnel to the greatest extent possible.

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Mr. J. A. Miller: I was just wondering if any tests had been run on operational shields in which black powder had been used?

Mr. Goff: Yes, we run tests on all operational shields at Lone Star. You'll see a paper included as part of the minutes of this meeting that will go into more detail. On these powders they are no longer what we thought they were. For example, say dextrimated lead azide of which we use many many pounds at Lone Star. We made such operational tests and found that a given amount of dextrimated lead azide at a given distance from a 1/2" plexiglass shield was entirely adequate to withstand the explosion of that amount of dextrimated lead azide. We now are using RD 1333 lead azide. We conducted a test and found that the exact same amount of RD 1333 lead azide in exactly the same location as was the dextrimated would blow the 1/2" plexiglass shield all over the place. So now it takes two people and we no longer trust this stuff. Our information was that this propellant wouldn't explode, but the shields came apart just the same.

## A STUDY IN DECOMPOSITION

By  
T. W. Driscoll  
Chemical Propulsion Division  
Hercules Powder Company  
Wilmington, Delaware

Between 1951 and 1958 at least five lives were lost in accidents within the explosives industry involving material which had been exposed to explosives but was thought to be either decontaminated or inert.

Hercules Powder Company was implicated in one of these accidents. This one occurred in Emmaus, Pennsylvania, on November 24, 1952.

Core rods which had been used in the production of rocket motors and shipped to an off-plant shop for Teflon coating were alleged to have been the cause of the incident. The cores had been subjected to a 400° decontamination cycle of eight hours prior to shipment and were considered safe.

Whether the accident was actually the result of an exothermic reaction from explosive contamination on or in one or more of the rods is not known. The incident was of a serious enough nature, however, to cause the Company to make extensive tests in the interest of establishing factual decomposition levels for various explosives which would permit reliable and nondestructive decontamination of tooling and equipment by means of controlled application of heat.

Work was begun on the premise that in the manufacture of solid propellant many pieces of equipment such as screens, mixers, dies, mills, cores, containers, hand tools, etc., are in intimate contact with explosive materials. Eventually, explosives may work into pores, interstices, crevices, corners or convolutes where removal by wiping or washing is difficult if not impossible. When contaminated items are to be placed in storage or may require shop work such as dressing, machining, welding or brazing, or if they are to be used in any way other than that for which they are normally used in propellant manufacturing, they must be considered hazardous. Hence, when the status of any contaminated item changes when it is to be worked on instead of with, the potential danger must be eliminated by some reliable process of decontamination.

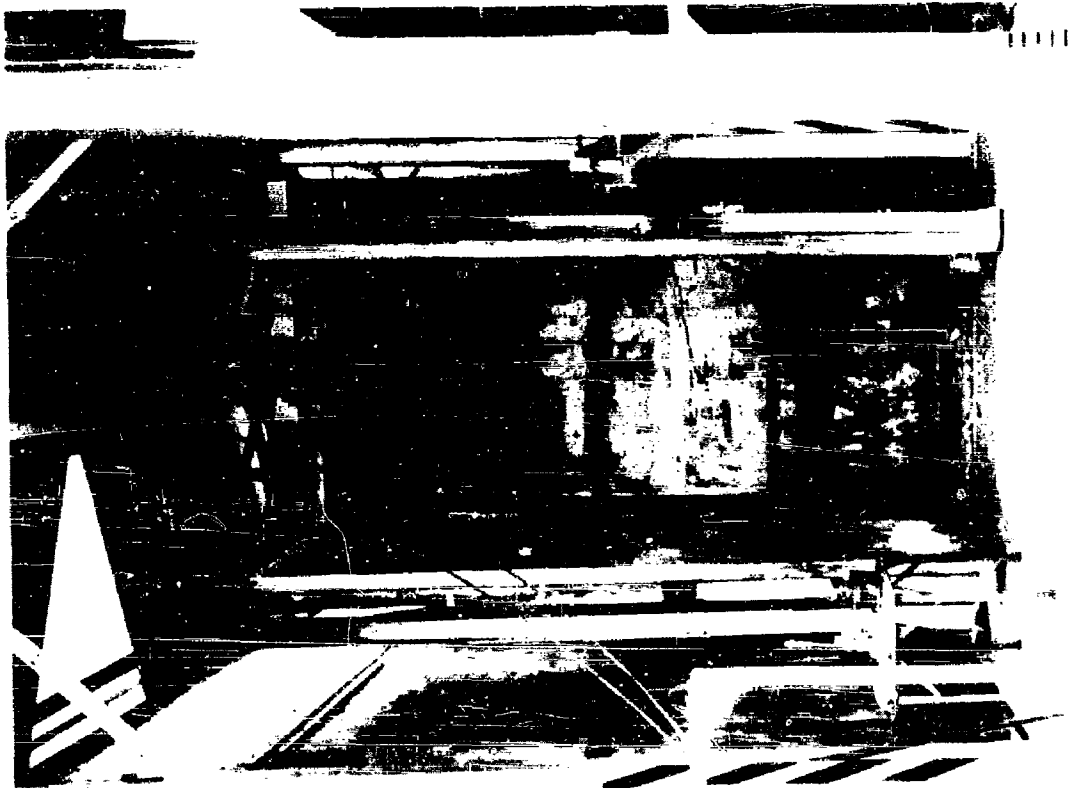
As used in this presentation, the terms "explosive" and "explosive material" will include any raw material, intermediate product or finished product which might detonate, explode or flash when raised to an elevated temperature, hammered, chiseled, machined or subjected to similar forms of shock or abrasion.

The purpose of the study which was made was to develop sufficient information for the promulgation of a standard decontamination procedure in which controlled heat would be used to effectively decompose residual explosive contaminants contained on or in any piece of metal equipment. It was found that little documentation of an informative nature existed covering the behavior of explosive materials under gradual increases of temperature and various conditions of containment. The study, then, was undertaken to determine answers to the following questions:

1. What is the approximate firing point or range in degrees F. of an explosive mass when the temperature of the atmosphere in which it is located is being gradually increased?
2. Does the physical form of the explosive (chunk, granular, film or fluid) influence the temperature at which ignition, flash or decomposition occurs?
3. Does an exposed explosive have the same ignition temperature as explosives that may be located within the body of the piece?
4. How is explosive material effected by a temperature slightly lower than ignition temperature, when exposed for a prolonged period of time?
5. Does the ignition temperature change when the rate of temperature rise is accelerated?
6. Does heat-transfer from air to metal occur at a constant rate and what is the differential between temperatures of air and metal surfaces at a practical rate of temperature increase of air?
7. How rapidly is heat transmitted from the surface of a large piece of metal to the center of the piece? At the heat acceleration rate used in this study, i.e., one to two degrees Fahrenheit per minute, what will be the greatest differential between surface and interior temperatures?

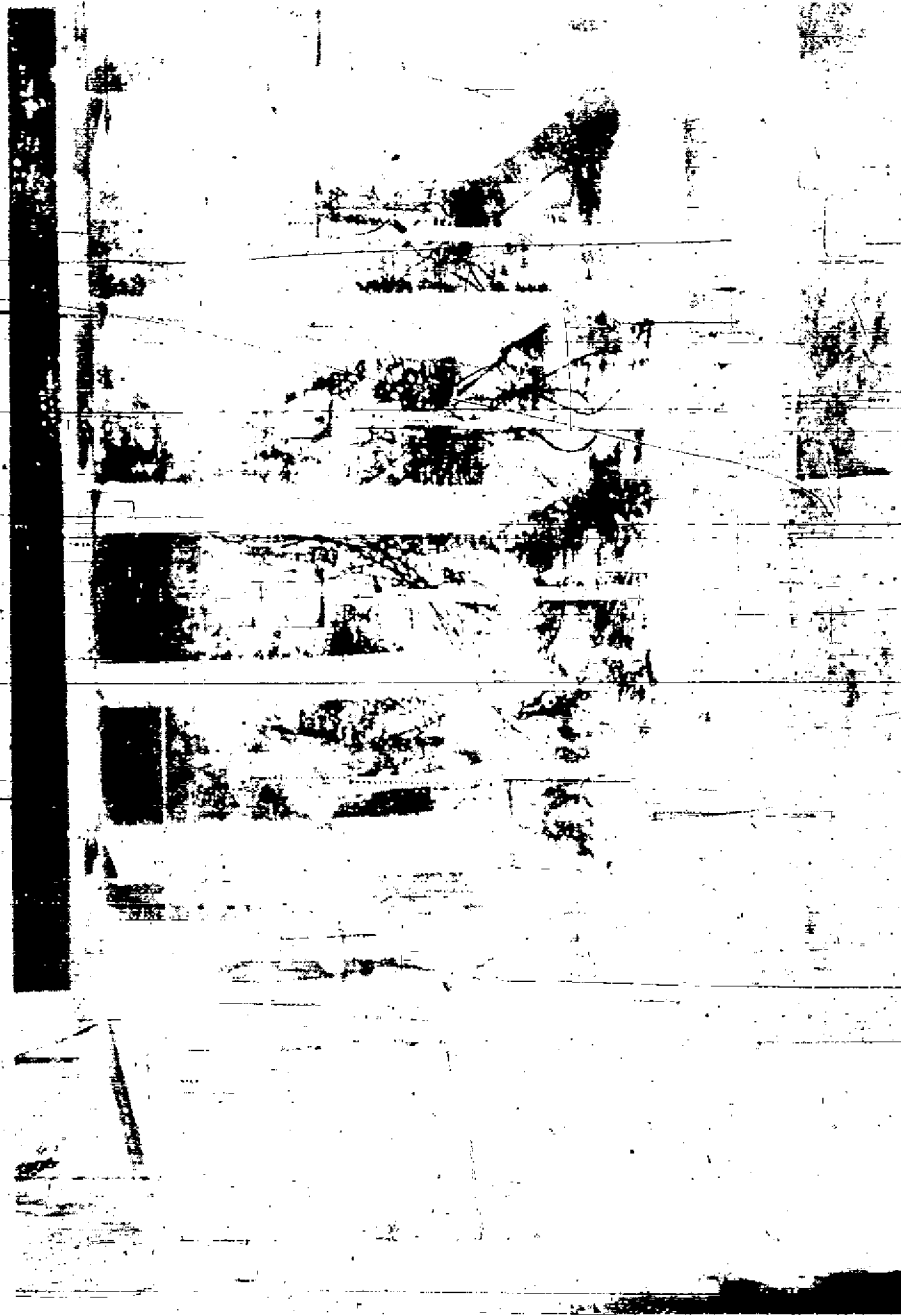
In undertaking a study of this type, the facilities to be used were of primary concern. It was considered important that a full scale oven instead of a small laboratory model be used in all tests. (SLIDE NO. 1) The interior dimensions of the oven used were six by seven by fourteen feet. Two doors formed the entire front of the oven. The doors and three walls were heavily insulated with block magnesia which was retained inside and out with heavy gage sheet iron. Inside the oven were several four-inch, perforated air dispersion pipes which distributed the hot gases from the burner house throughout the oven. (SLIDE NO. 2) Halfway up one wall of the oven an iron-constantan thermocouple was located which actuated a Minneapolis-Honeywell recorder and controller for the Kemp butane burner located about one hundred feet behind and twenty feet above the oven. Butane gas was fed to this burner and the hot gaseous products of combustion were driven into the oven through a heavily insulated duct. Temperatures were controlled automatically by using operating cams in the controller, or manually by manipulating the rheostat controller on the equipment. The highest practical temperature maintained in the oven was 750°F.





Full Scale  
Decontamination  
Oven

# Interior Of Oven With Test Trays



For the purpose of the decontamination study, additional indicating, signalling and recording devices were installed in the burner house. More detail regarding this equipment may be had on request.

Six pedestal mounted, portable trays were installed in the oven to hold the explosive samples and leads for the safety firing, signalling, indicating and recording devices located in the oven. (SLIDE NO. 3) Each tray consisted of a 6" x 24" transite board on which was mounted a sheet metal retaining fence 4" wide by 16" long by 3/4" high. Within the fence of each tray was a safety firing device consisting of two terminals 10 inches apart which supported a 12-inch piece of nichrome wire. The nichrome wire was connected to a secondary transformer winding on top of the oven. When the circuit was closed, the wire, which has a resistivity of about 110 compared to aluminum with a resistivity of about 2, became white hot. This gave the safety firing device a two-fold function: the ability to destroy any explosive which might be left on the tray prior to opening the doors and also to test the signalling devices for proper operation.

The signalling devices were installed at one end of the trays. These consisted of two stainless discs about 2 inches in diameter and a half-inch thick. One disc was held approximately one inch above the other by means of a cotton thread which was secured at one end of the tray, ran through the tray slightly above the nichrome wire, under a keeper bar, over a guide bar to the top disc. The explosive to be tested was placed on the nichrome wire just below the cotton thread. When the explosive ignited, the cotton thread was severed, dropping the top disc to the bottom disc and closing a six-volt circuit which led to the signal buzzer and light on the instrument panel in the burner house. Each tray was connected to a light and buzzer the number of which corresponded to that of the tray.

Each tray was also serviced by an individual thermocouple which led to a Leeds and Northrup Micromax recorder in the burner house through a point on an eleven-point selector switch thus permitting any one of the thermocouples to be connected to the recorder as desired.

To check the accuracy of the recording devices and thermocouples, a reference assembly was used. This assembly consisted of a one liter distillation flask heated with an electric hot plate and a thermocouple permanently suspended in the steam which issued from the flask. The flask and hot plate were installed just outside the doors of the oven. During the course of the study, it was found that when the selector on the recorder was switched to the thermocouple suspended in steam, the recorder pen moved rapidly to the 210 degree F. line. Any defect in the instrumentation would have been indicated by a departure from the 210 degree recording of the reference thermocouple. Furthermore, any idiosyncrasy of the other six thermocouples which were used would be shown by erratic breaks in what should be a solid line.

Another valuable function of the indicating and recording devices was the automatic recording of the firing point of the explosive being tested. When the suspected firing point was approached, the operator stood by the instrument board and as soon as the light and buzzer indicated a tray had

Reference Assembly  
For  
Checking Accuracy  
Of  
Recording Devices  
And  
Close-up Of  
Test Trays



fired, the selector was immediately switched to the number corresponding to the tray that signalled. Since the thermocouple on that tray was covered by the explosive, the recorder pen moved across the chart as soon as the explosive fired, leaving a horizontal line almost at right angles to the temperature track of the trays. The point of departure from the vertical could then be read as the firing temperature of the explosive being tested.

The study proved conclusively that controlled heat applied in the proper way in the right kind of equipment is a reliable means of decontaminating. It was found that the answers to those questions previously mentioned were as follows: (SLIDE NO. 4)

1. At a temperature rate of rise of one to two degrees per minute, it was found that the firing point for those explosives tested fell within the following ranges in degrees F:

Single, double, and triple base propellants and casting powder - 300 to 325. Several formulations of single base were tested and firing points were found throughout this range. Firing points for double base formulations were found to be in the top half of the range and firing points for triple base and casting powder formulations were mid-range.

Nitro cotton - 330 to 345.

Nitroglycerin - 315 to 325, casting solvent - 300 to 315. When these substances were tested as a thin surface film on a piece of steel, they volatilized or decomposed before the temperature reached the firing point for a greater mass of these explosives. When closely confined and heated by increasing the ambient temperature, these materials began to decompose with liberation of heat and the production of gases which greatly increased the pressure. In the neighborhood of 300° F. decomposition of nitroglycerin is extremely fast. It was found that pure nitroglycerin will, if hermetically sealed within a pocket, explode at about 315° F. On the other hand, casting solvent with a firing point of 300°F. did not explode but created sufficient gas to rupture the test vessel by pressure.

Pentolite - 330 to 340.

Black Powder - 460 to 480.

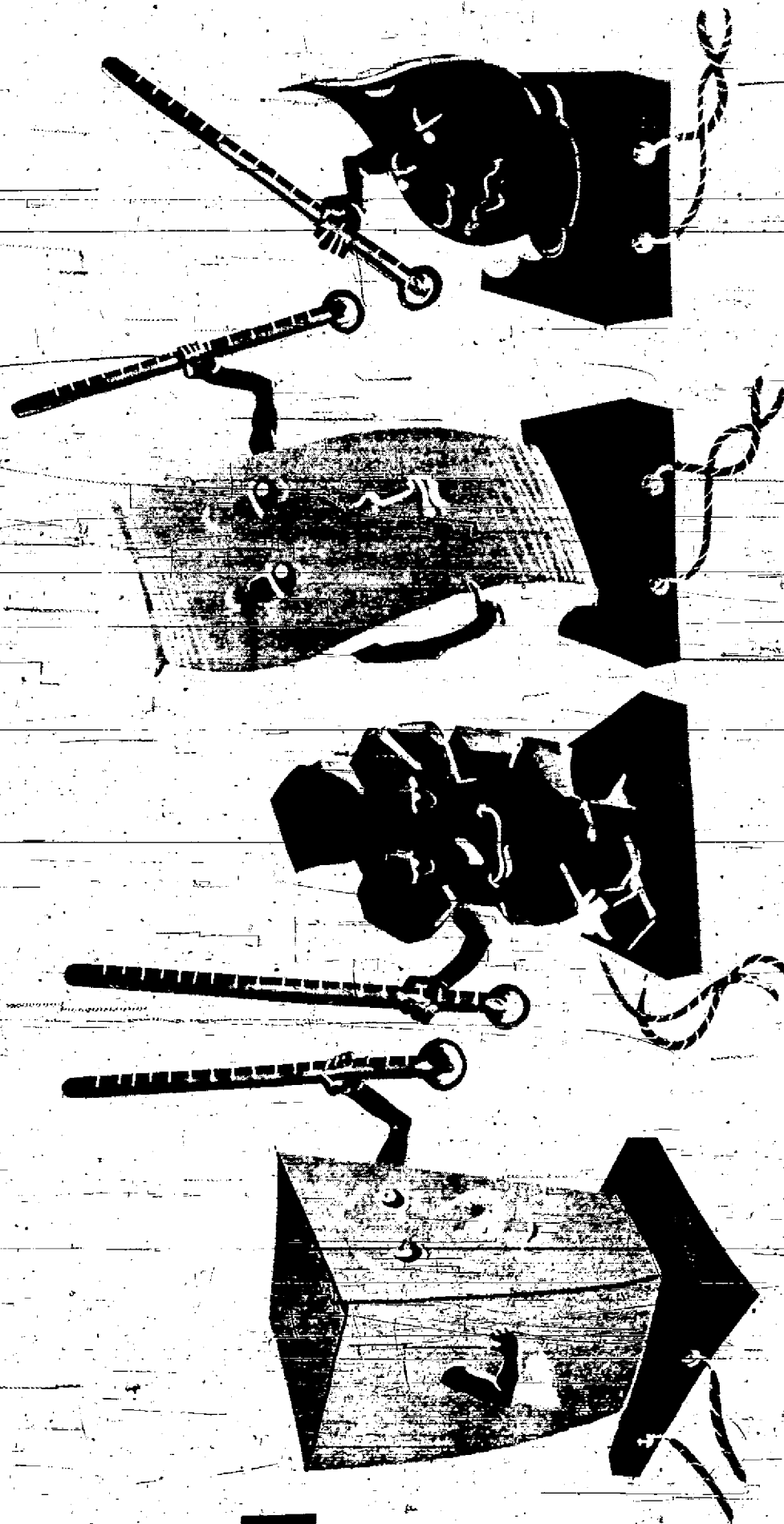
Nitroguanadine - 460 to 490.

TNT, RDX, HMX, AP - 540 to 560. (SLIDE NO. 5)

(When RDX and HMX are combined, exothermic reaction will occur at approximately 325°F.)

2. In testing explosives in various physical forms, it was found that the temperatures at which exotherms were reached remained constant. Extraneous substances such as graphite, CA, floor sweepings, grease, etc. were mixed with the explosives and in no case was it found that such admixtures raised the ignition point of the contaminant. (SLIDE NO. 6)

2..... Does the physical form of the explosive (chunk, granular, film or fluid) influence the temperature at which ignition, flash or decomposition occurs.....?





• Does an exposed explosive have the same ignition temperature  
as explosives that may be located within the body of the piece • • •

3. Explosives were placed in all manner of inaccessible locations and the decomposition temperature was found to be in the same range in all instances. (SLIDE NO. 7)

4. Effective decomposition does occur at temperatures slightly below (20-30 degrees F.) the established firing point for a solid explosive. Sixty to seventy hours were required to reduce the explosives tested to a harmless carbonaceous residue. This lengthy time cycle would tend to make such a process highly impractical from both an economical and utilization standpoint. These tests did verify, however, that caution must be exercised in establishing temperatures and time cycles for high temperature curing processes. (SLIDE NO. 8)

5. The rate at which the temperature of the piece being decontaminated is increased from the starting temperature to the decontamination temperature has no effect on the firing point of the explosive so long as the period of time required to reach the decontamination temperature is between 3 and 7 hours. (SLIDE NO. 9)

6. The rate of heat-transfer from a gaseous ambient to a solid is a function of the rate of temperature increase of the ambient. If the rate of increase of the ambient is constant, the differential between the ambient and the solid will also be constant. If the rate of increase of the ambient is not constant, the differential will depend on several factors such as temperature, the rates of increase and the composition of the solid. Except where carefully controlled conditions are maintained, there is no predictable correlation between the air temperature of the oven and the temperature of the metal part being decontaminated. Tests showed that at 80°F. the air and metal were the same temperature. By the time the temperature of the metal had reached 90°F., the air temperature had risen to 155°F. In the tests which are discussed here, the greatest differential was after 4-3/4 hours when the temperature of the metal was 260°F. and air was 370°F. (SLIDE NO. 10)

7. Conduction of heat from the surface of a metal piece to the center is also related in some degree to the rate of temperature increase and intensity. In these tests the greatest differential between surface and interior temperature was 12°F. with the greatest time lag being 15 minutes.

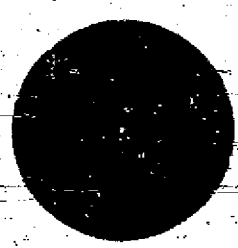
It is not expected that all decontamination work will be done in an oven. Preliminary to the application of heat, procedures call for the contaminated area of the item to be washed with an appropriate solvent and/or soap and water thereby removing most, and in some case all, of the contaminant. Thus the heat treatment may be considered the finishing operation or positive assurance that the last trace of explosive has been removed and the metal part is rendered safe for handling or being worked on. It is not implied that all items must be subjected to heat, either in an oven or by flashing, to be considered decontaminated. Certainly some materials can be thoroughly decontaminated by cleaning with the proper solvents, which may include soap and water, or chemicals, after some exposures.



4

How is explosive material effected by a temperature slightly lower than ignition temperature when exposed for a prolonged period of time . . . . .





5

..... Does the ignition temperature change  
when the rate of temperature rise is accelerated .....

?

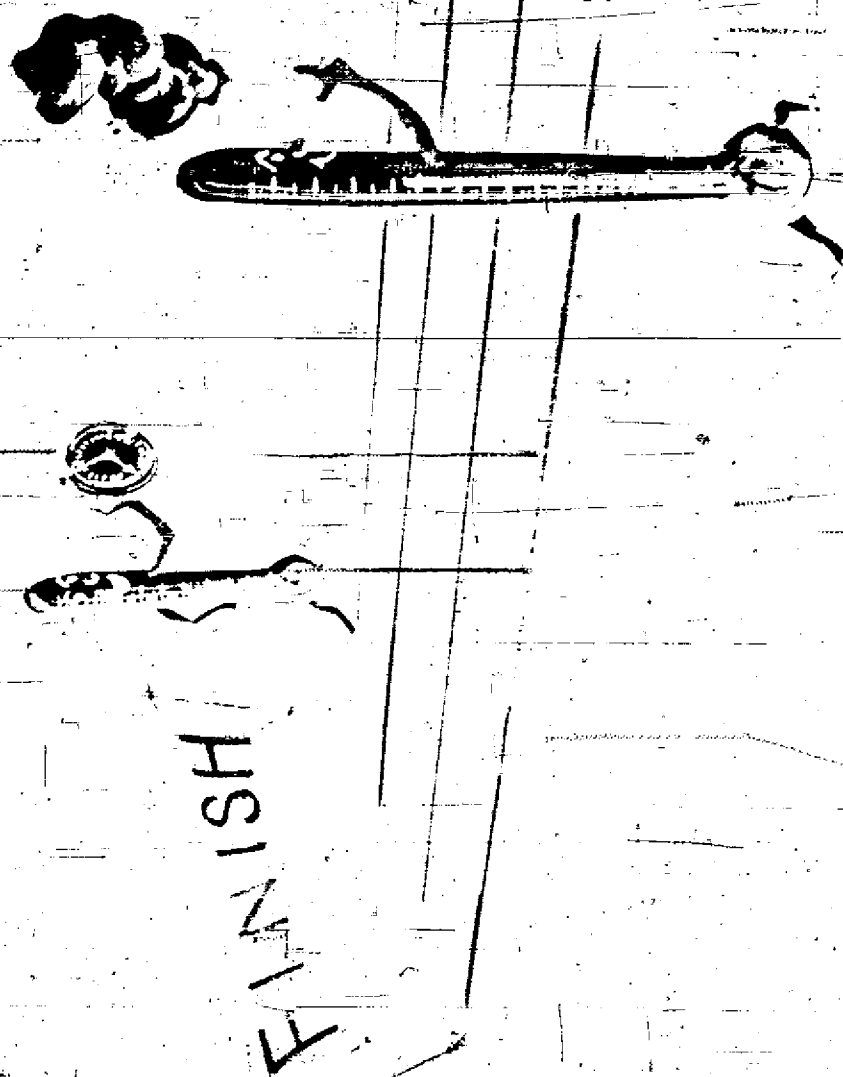
6

Does heat transfer from air to metal occur at a constant rate and what is the differential between temperatures of air and metal surfaces at a practical rate of temperature increase of air . . . . .



7...

How rapidly is heat transmitted from the surface of a large piece of metal to the center of the piece ? At the heat acceleration rate used in this study, i.e., one to two degrees Fahrenheit per minute, what will be the greatest differential between the surface and interior temperatures . . . . .



From information which is available, it is indicated that controlled heat decontamination is a requirement in all cases where explosive contaminants may be held within the interstices of metal or larger void areas that are beneath the surface of the material from which a piece of equipment or related component is manufactured. Material to be decontaminated by heat normally will be metal. Such materials as wood, plastics, rubbers, asbestos, etc. usually will be ruined by the decontamination process or would have insufficient reclaim value to justify the expenditures of time and money required to render them safe.

It seems well justified to assume that any material which is known to have been in contact with a liquid explosive (such as nitroglycerin), whether by intent or accident will be adequately decontaminated only after it has been maintained at a temperature of not less than 450°F. for four hours. The "temperature" referred to here is the temperature of the material that is being decontaminated, not the temperature of the air surrounding the material.

Further, adequate decontamination will not be considered as having been accomplished unless there is continuous monitoring and recording of the temperature of the item being decontaminated when the sensing element is located on the most massive portion of the piece of equipment or component. If elongated or complex items are involved, it may be necessary to utilize several sensing elements.

In 1945 War Department Supply Bulletin 5-52, DECONTAMINATION PROCEDURES, was published. It is suggested that this venerable document, used by all services as the "bible" for decontamination, be revised to reflect newer techniques and provide factual information regarding decomposition characteristics, ignition temperatures, etc. of commonly used contaminants. SB 5-52 recognizes no method of explosive decontamination other than "flashing". It is submitted that the application of controlled heat for an adequate length of time at temperatures known to be above the effective decomposition level of the explosive involved is a more reliable, scientific, and economical method of decontamination than subjecting material to flame for an undetermined period of time at unknown temperatures.

Supply Bulletin 5-52 implies that various degrees of decontamination may be attained. Would it not be far more realistic to consider the three "X" code as warning that contamination is very possibly present, instead of a symbol which is conducive to adopting a false sense of security?

No doubt other companies have developed procedures for decontamination not described in SB 5-52. Hercules Powder Company would appreciate hearing of any method by which to reliably accomplish this necessary operation.

Mr. Tweed: You mentioned that RDX and TNT had values above 500° F.  
Is that correct?

Mr. Driscoll: This is what we found.

Mr. Tweed: Have you ever tried mixing the two together?

Mr. Driscoll: No, I'm sure that we haven't.

Mr. Tweed: It might be of interest to know that when you mix the two together and put a few additives to make it Comp. B you can get the material to explode at temperatures of between 325° and 350° F. This was done in a 2 ounce sample and also in 155mm shell which has 15 pounds.

Mr. Jezek: On this Supply Bulletin SB 5-52, for the benefit of those who are in the military, five years ago we revised this publication and since the Ordnance Corps did not sponsor it we had to send it to the Corps of Engineers for publication. The Corps of Engineers promised us that they would reprint this bulletin. However, during the interim period they came out with a circular and said this book is now obsolete. It has been rescinded. We have tried on several occasions to have this book republished. But we haven't been able to do it. Why they don't do that I don't know. Insofar as the Army Materiel Command is concerned, on decontamination, we agree that if you need or have to re-use equipment that is contaminated, we should heat it and get the explosives out. However, we can't see going to using ovens and all that sort of paraphernalia, decontaminate piping, etc. that is taken out of our buildings. It is too expensive so we just set it on some dunnage and ignite the dunnage and let it go. If it blows it blows, if it doesn't then we have a resale value for the scrap.

PLANNING AND SITING FOR THE  
MANUFACTURE OF LARGE SPACE BOOSTERS

Carl Wiuff  
Aerojet-General Corp.  
Homestead, Fla.

Mr. Wiuff presented a series of slides showing the planning and siting of the new Aerojet Dade County, Florida Plant.

AEROJET DADE COUNTY PLANT

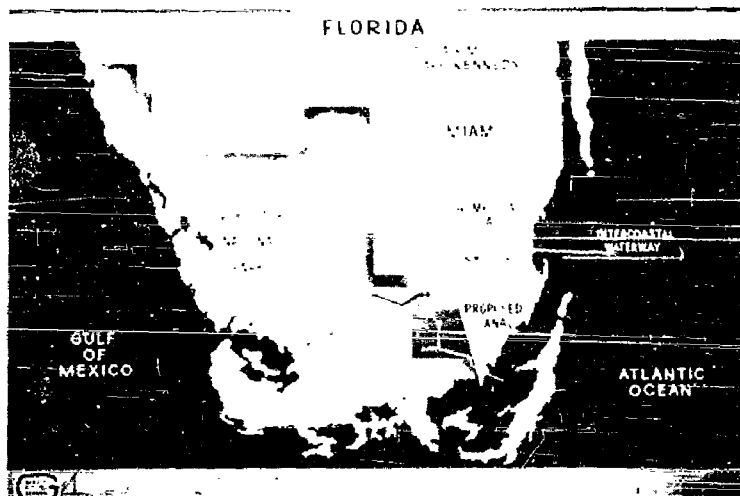


Figure 1 - Location in Florida of the Dade Division  
75,000 acre Plant of Aerojet-General Corporation

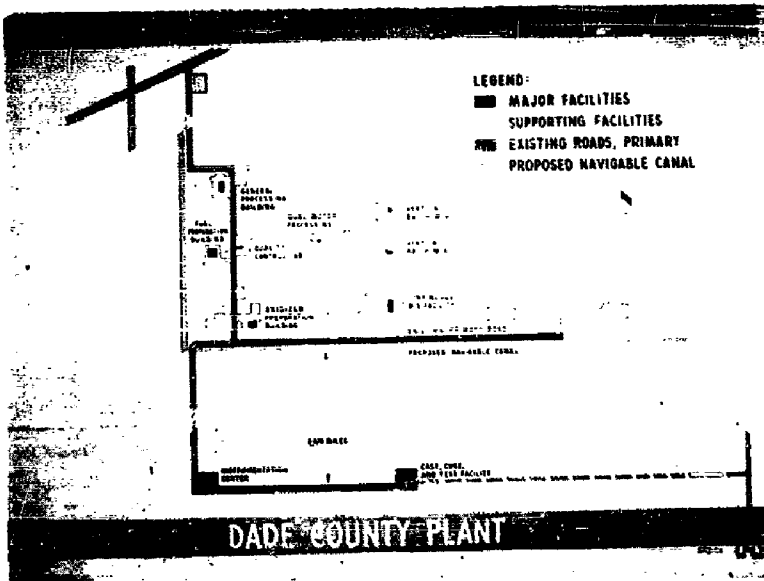


Figure 2 - Building siting of Aerojet Dade Div. Plan



Figure 3 - General purpose building used for chamber preparation and liner installation



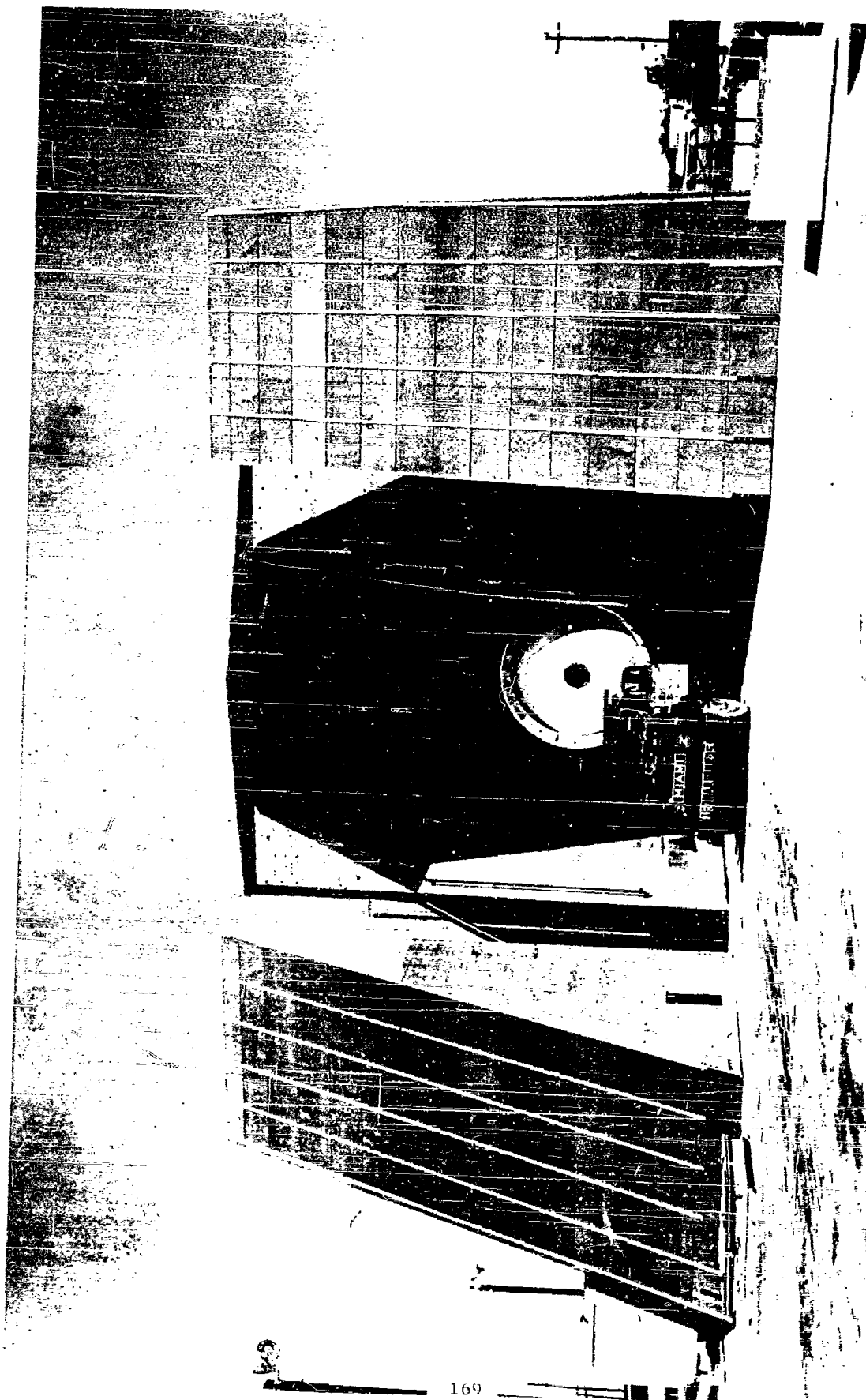


Figure 4 - Placing 120" chamber in general purpose building

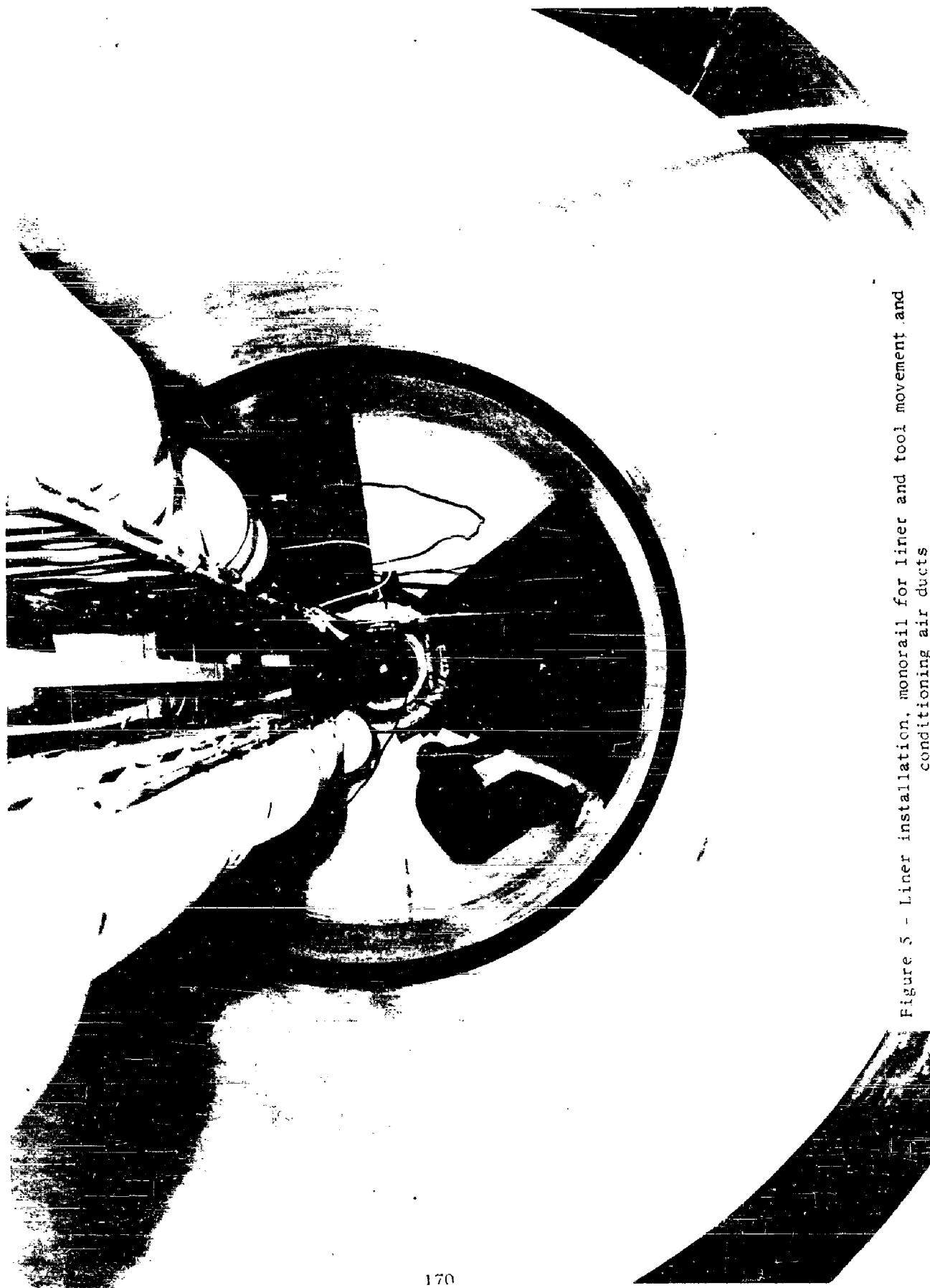


Figure 5 - Liner installation, monorail for liner and tool movement and conditioning air ducts



Figure 6 - Abraising liner, air tools and air masks

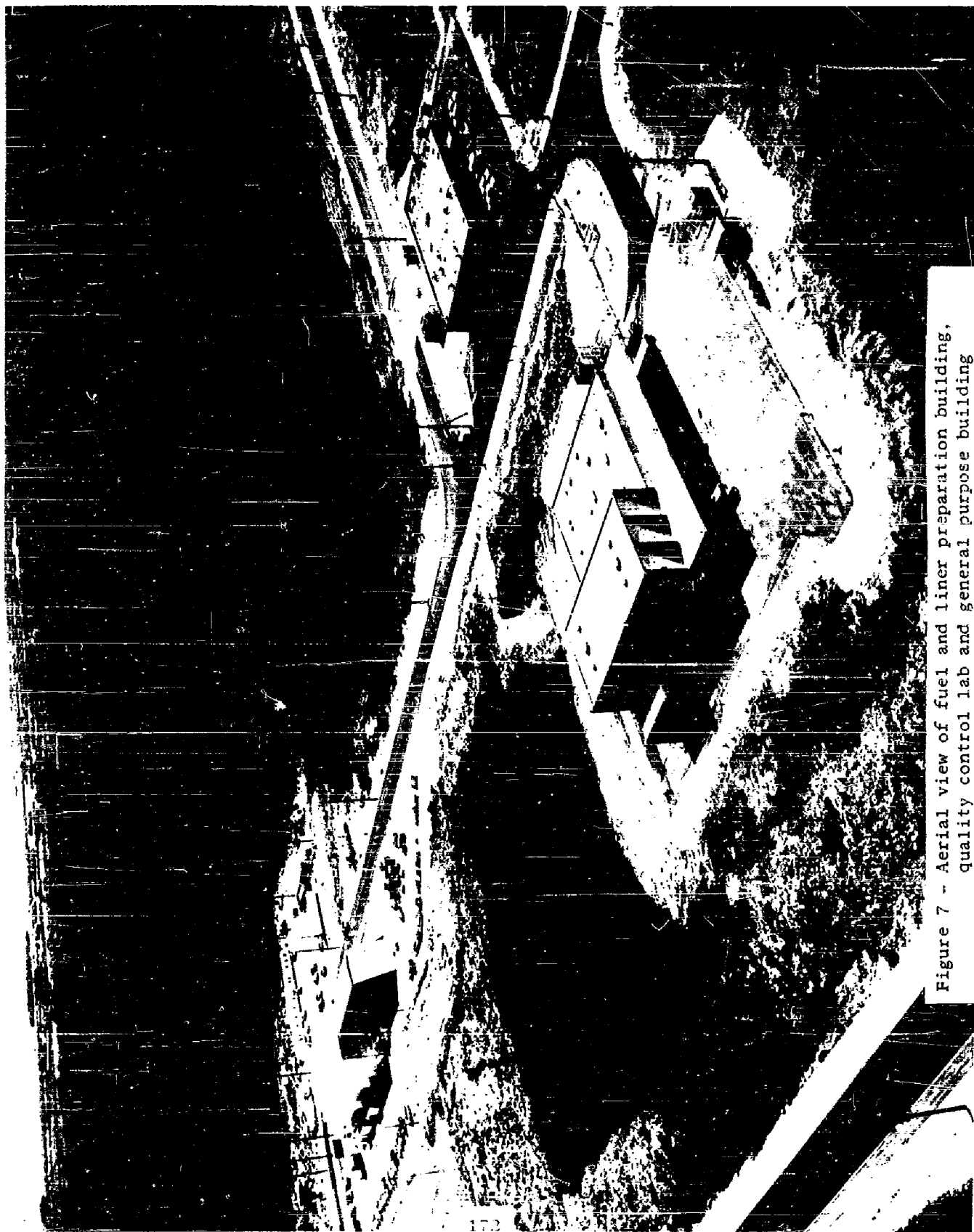


Figure 7 - Aerial view of fuel and liner preparation building, quality control lab and general purpose building

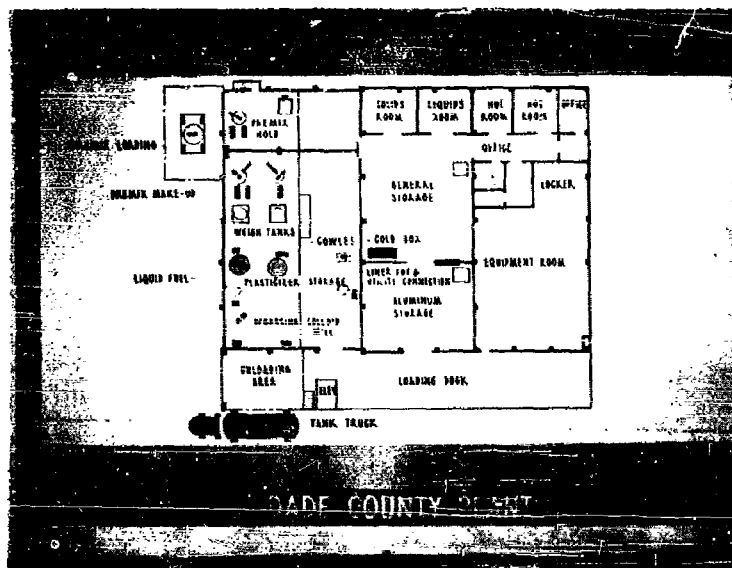


Figure 8 - Fuel and liner preparation building layout

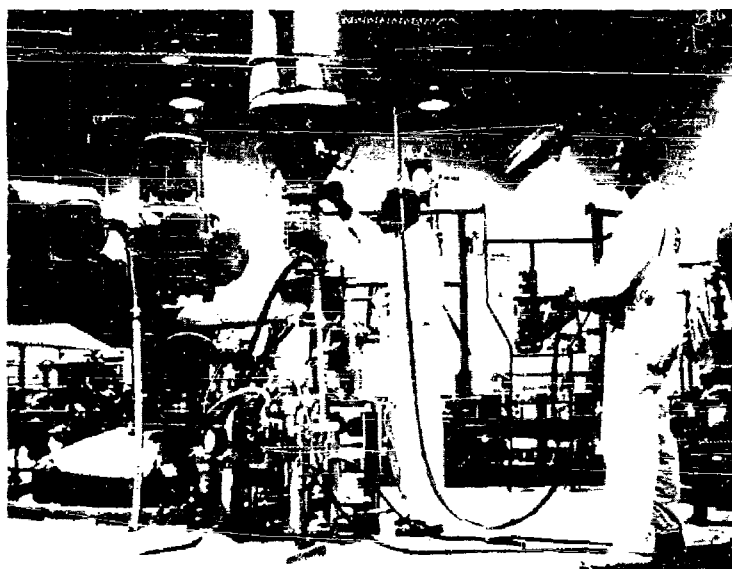


Figure 9 - Aluminum powder addition to pre-mix



Figure 10 - Aerial view of oxidizer preparation building, in foreground.

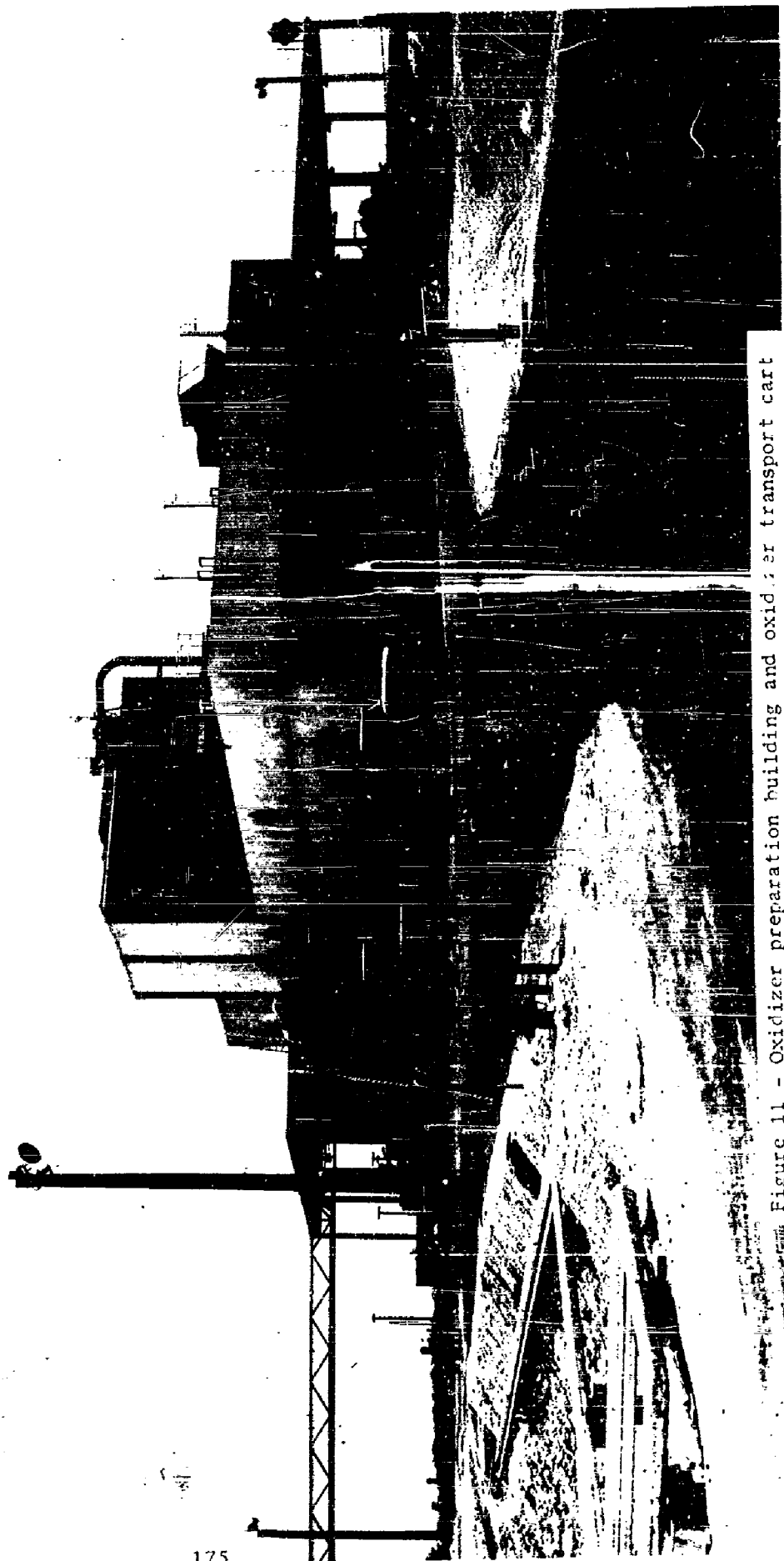


Figure 11 - Oxidizer preparation building and oxidizer transport cart

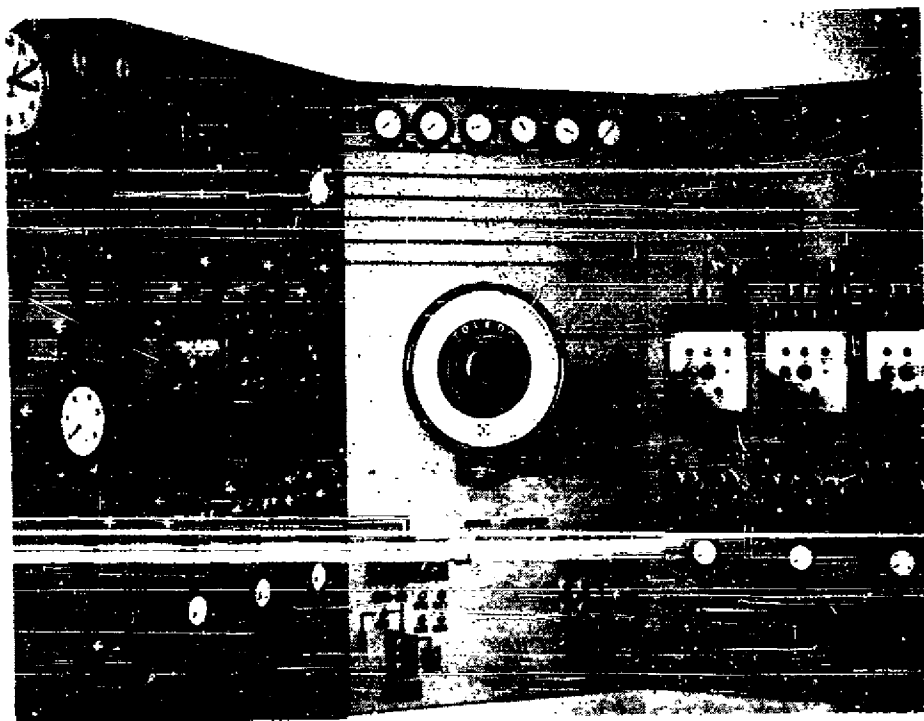


Figure 12 - Control room of oxidizer prep building





Figure 13 - Receiving platform for 5000 pound oxidizer tote bins



Figure 14 - Hydraulic tote bin dumpers



Figure 15 - Aerial view of vertical batch mixing complex

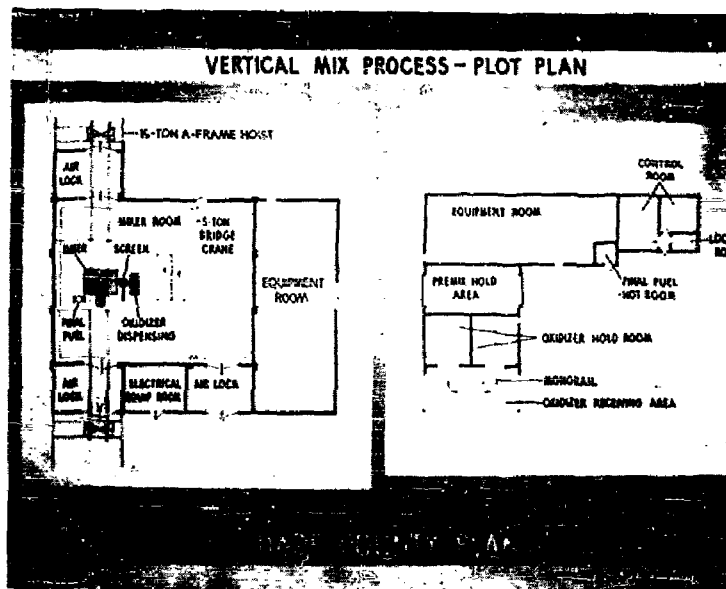


Figure 16 - Vertical mix building layout

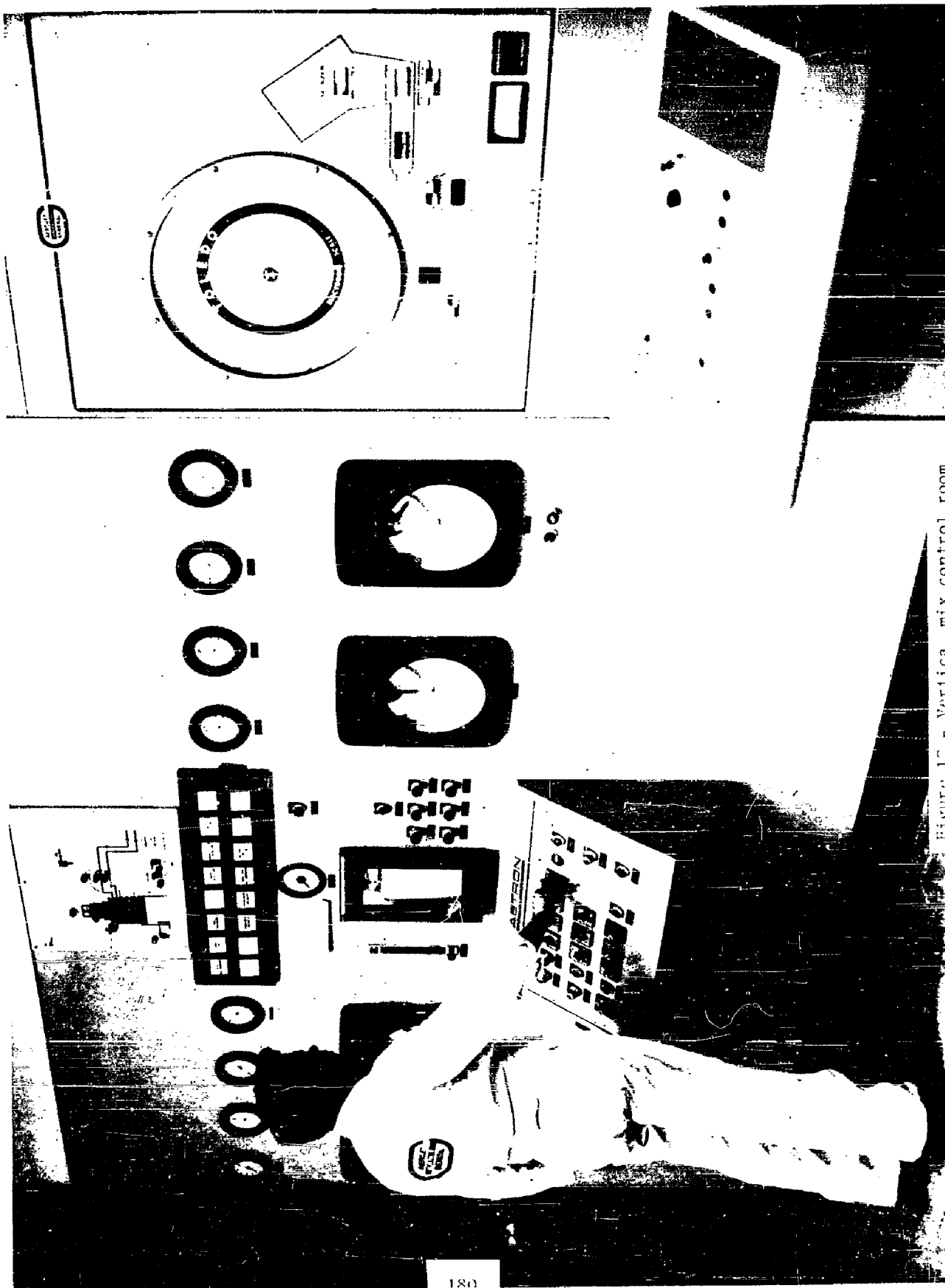


Figure 17 - Vertica, mix control room

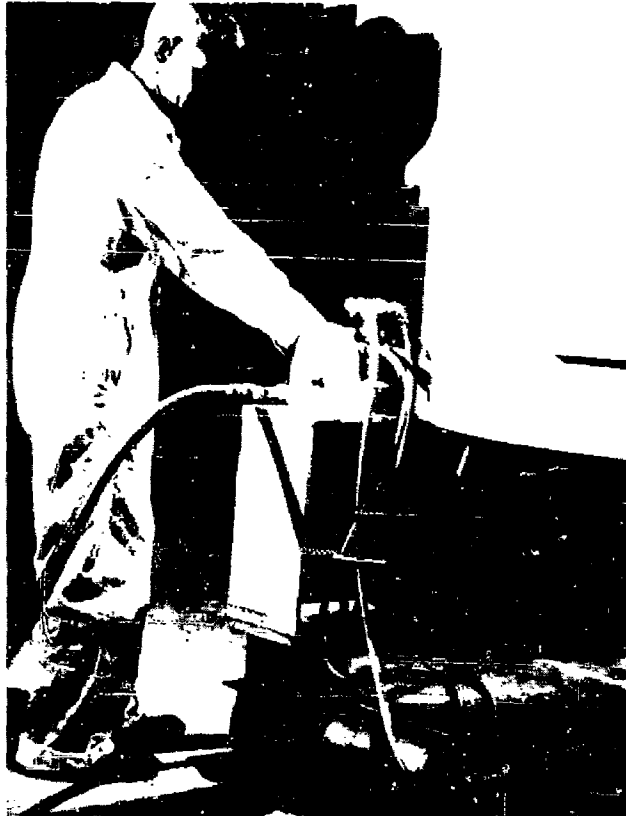


Figure 18 - Air motor drive on all propellant and ingredient transport vehicles

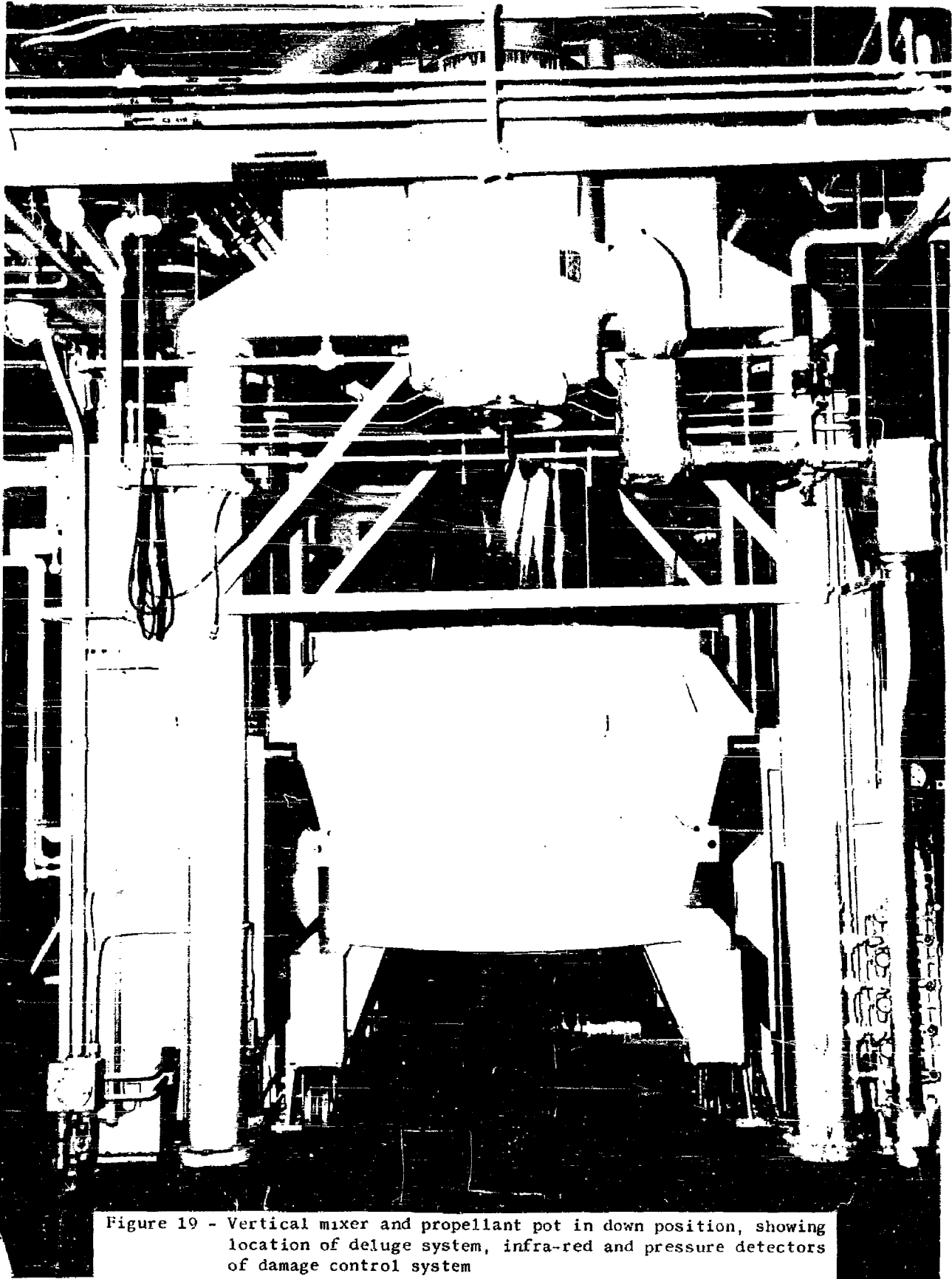


Figure 19 - Vertical mixer and propellant pot in down position, showing location of deluge system, infra-red and pressure detectors of damage control system



Figure 20 - Aerial view of continuous mix facility

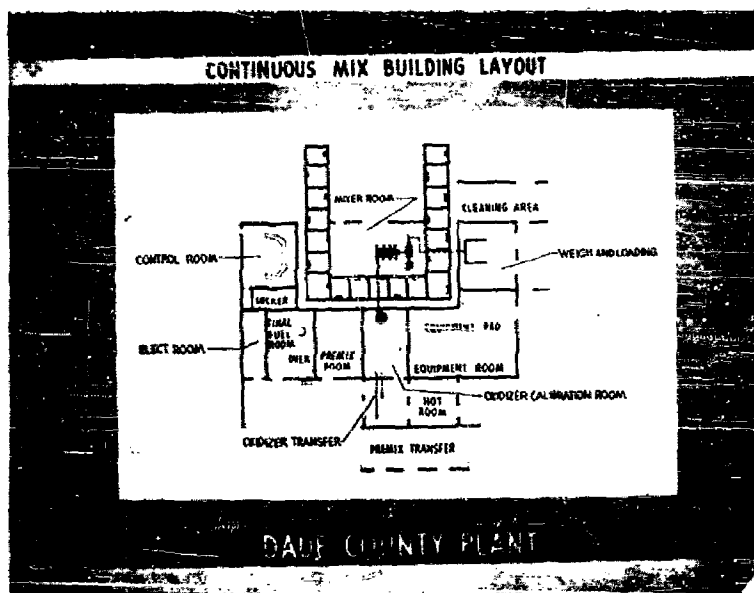


Figure 21 - Continuous mix building layout



Figure 22 - Receiving pre-mix from fuel preparation building at continuous mix



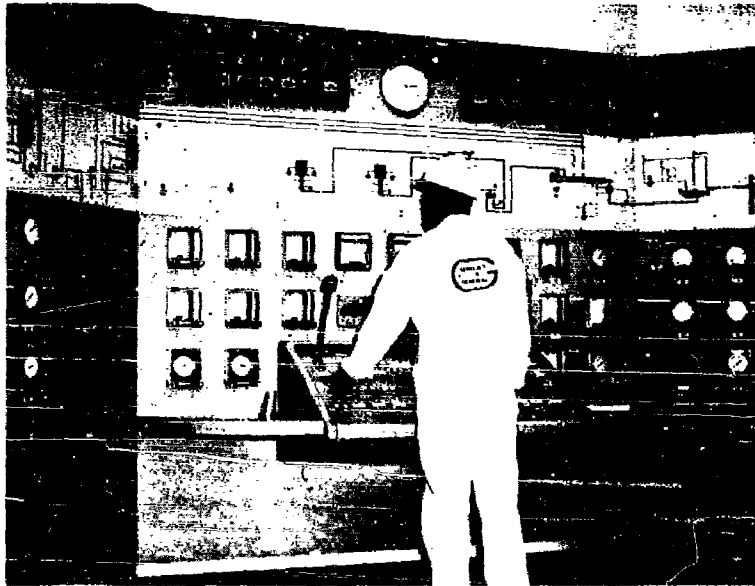


Figure 23 - Continuous mix control room

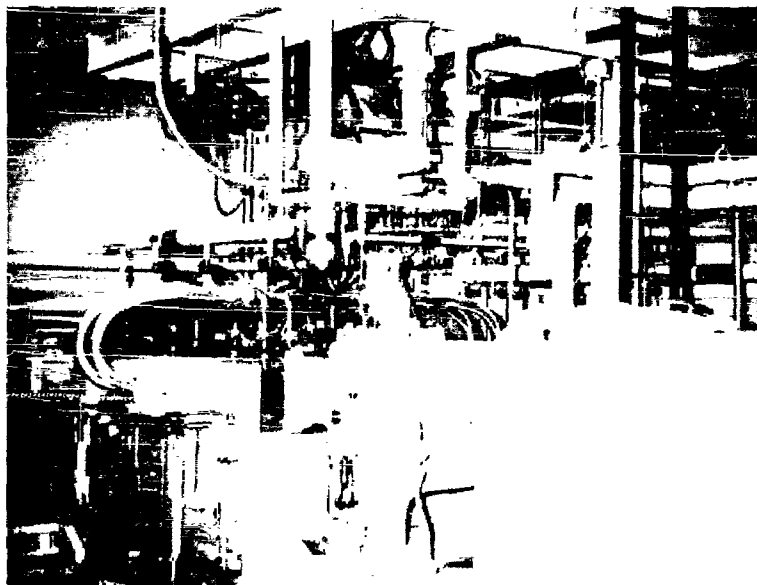


Figure 24 - Continuous mixer

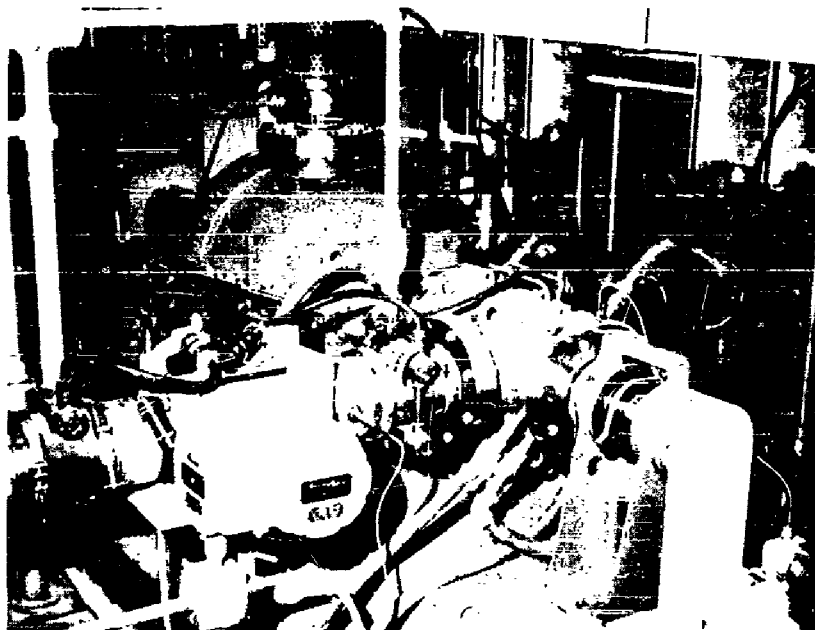


Figure 25 - Surge pot, de-aerators, roto feed,  
accurray and damage control detectors  
and devices

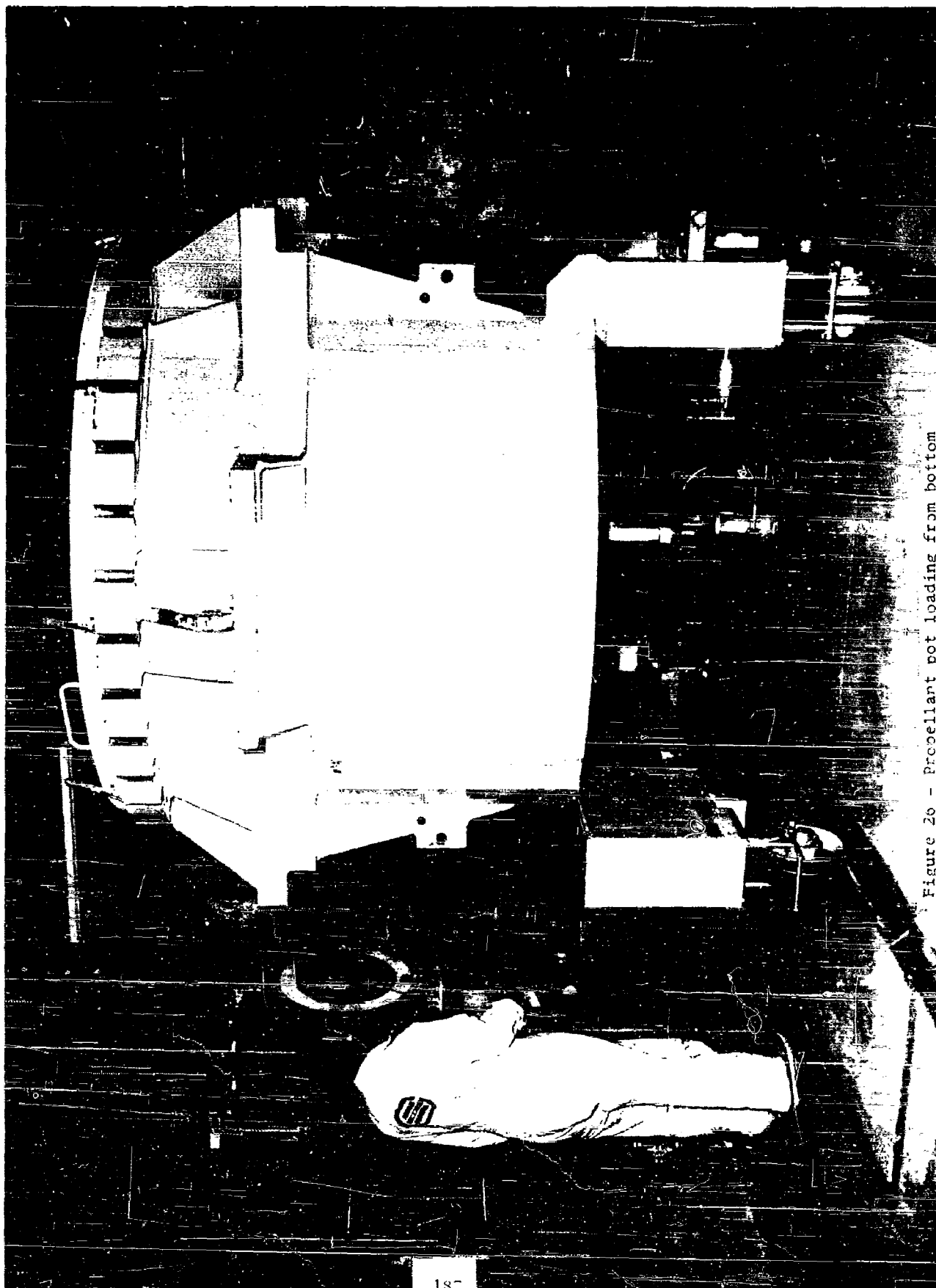


Figure 26 - Propellant pot loading from bottom

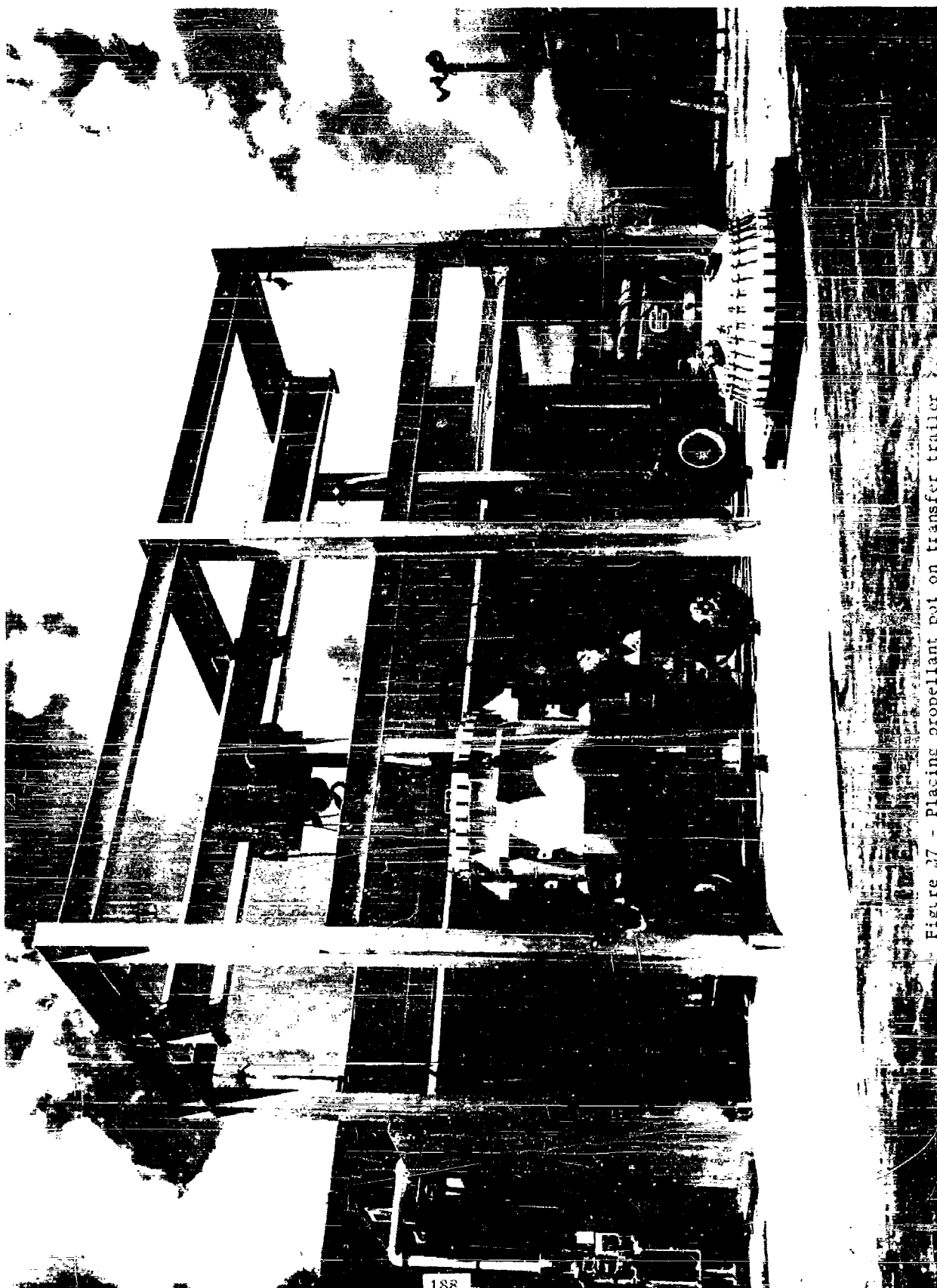


Figure 27 - Placing propellant pot on transfer trailer



Figure 28 - Qualification motor process building, foreground. Cleaning building,  
center. Water pumping station, top. Continuous mix facility,  
upper left



Figure 29 - Upper level sample draw-off to lower level quality process building



Figure 30 - Aerial view of instrumentation building, foreground.  
Cast, cure and test facility, background.

# BASIC CAST, CURE AND TEST FACILITY

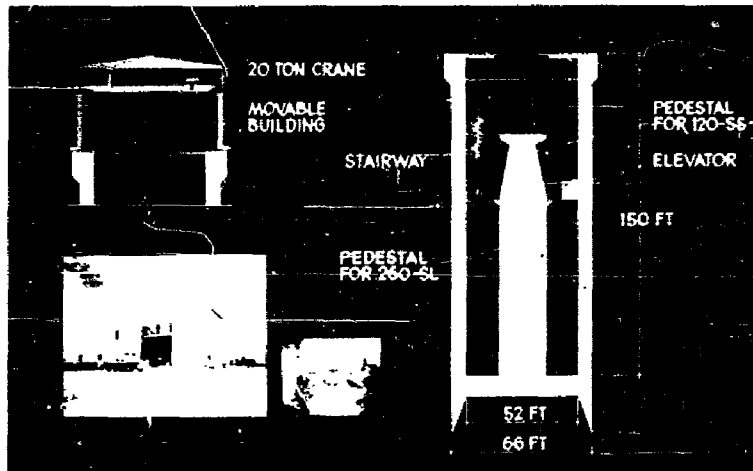


Figure 31 - Basic cast, cure and test facility

## 120-SS-1 MOTOR CASTING

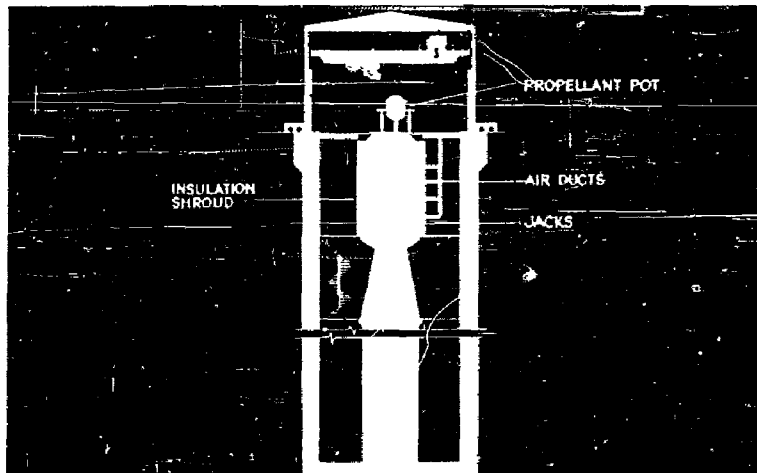


Figure 32 - 120" motor casting





Figure 33 - Building moved prior to placing 120" chamber in caisson

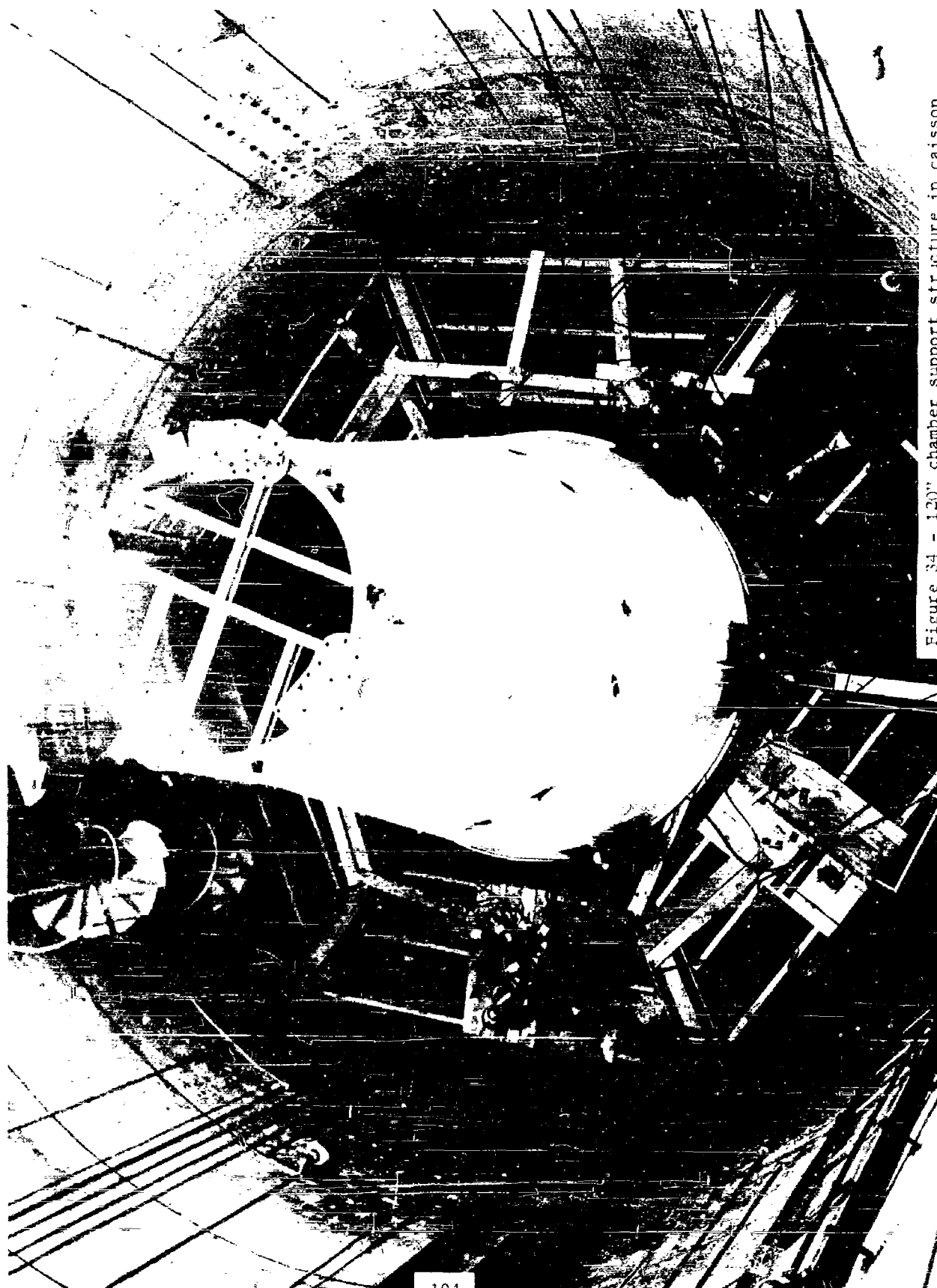


Figure 34 - 120" chamber support structure in caisson

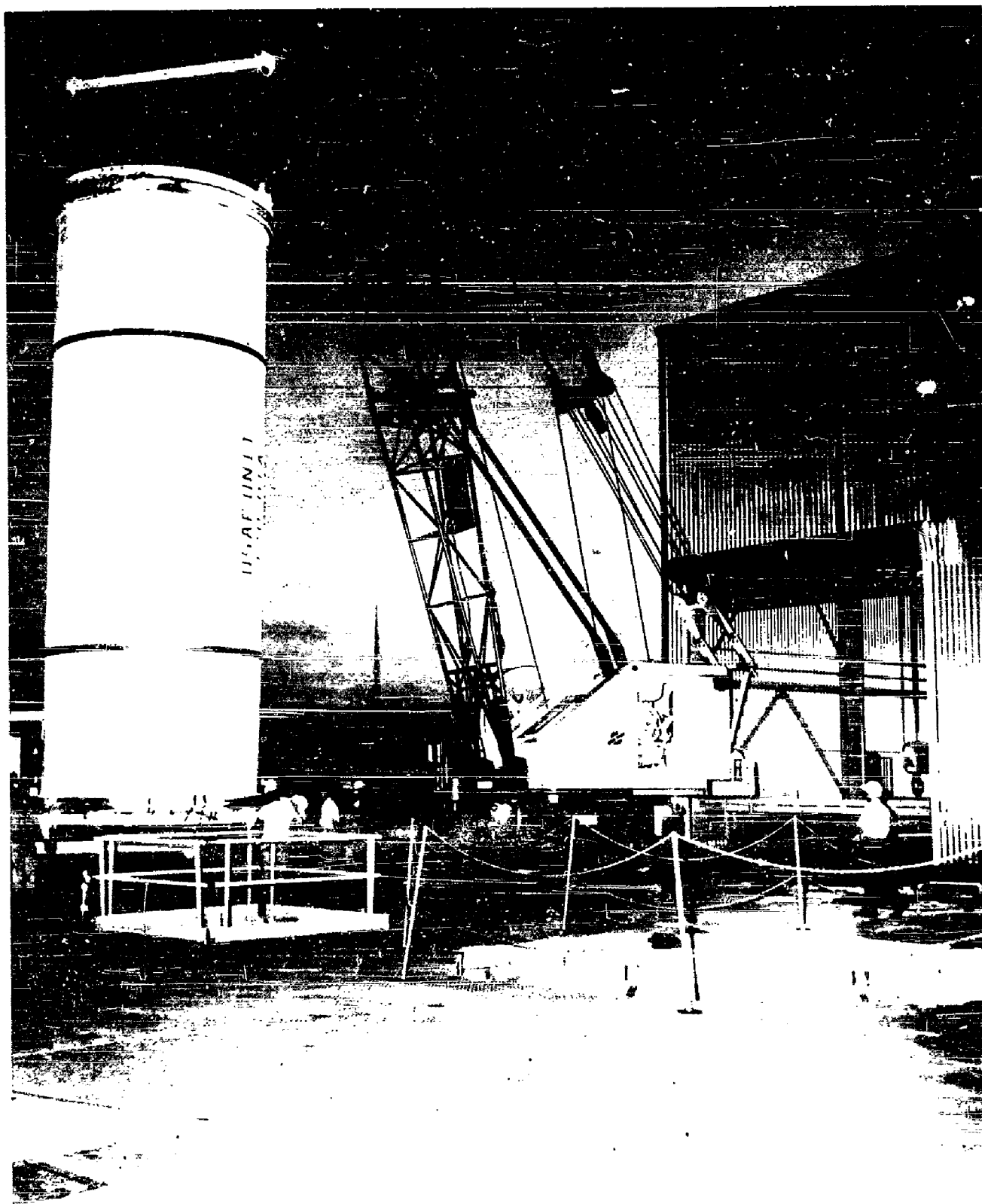


Figure 35 - Placing 120" chamber in caisson

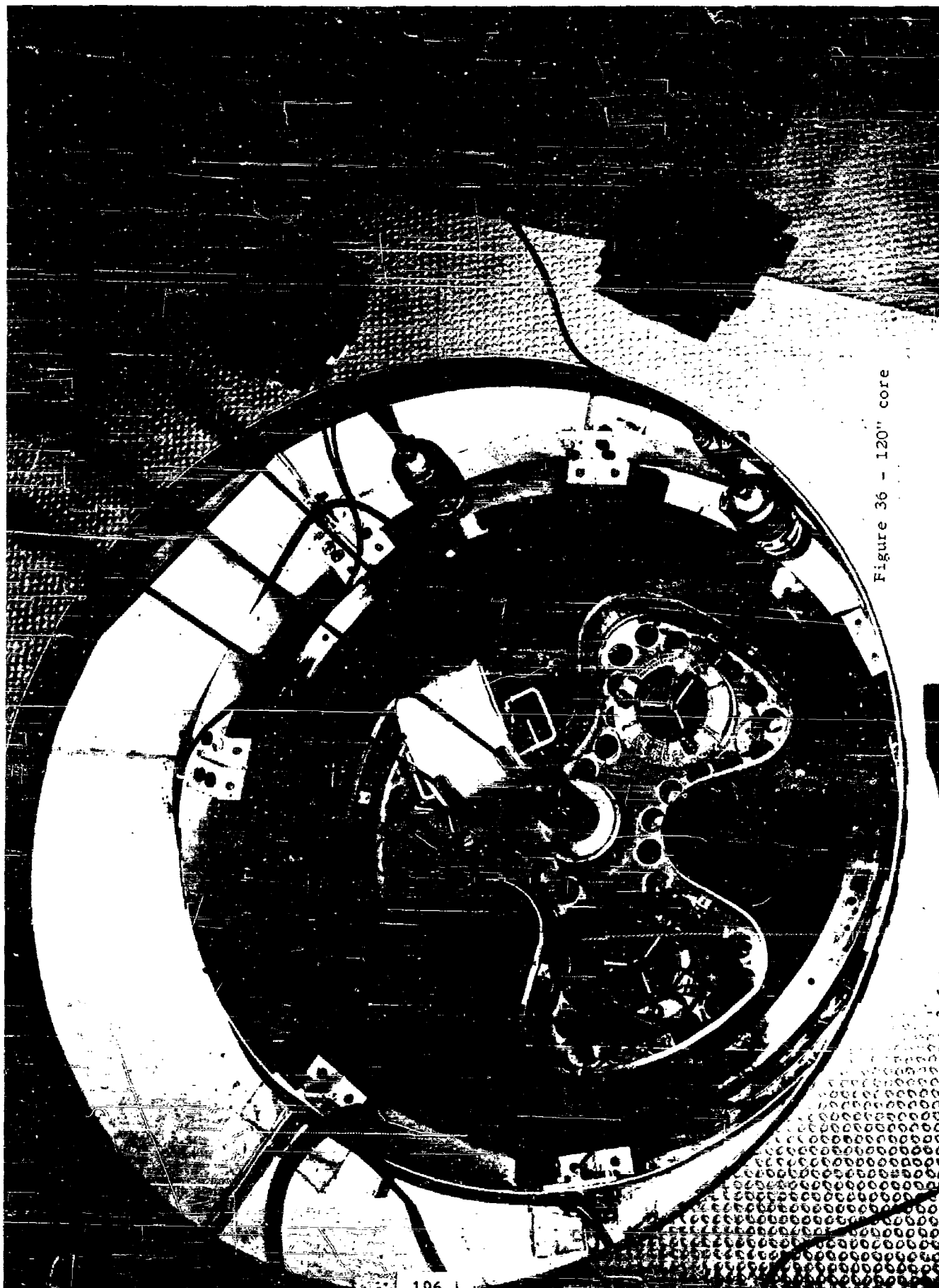


Figure 36 - 120" core

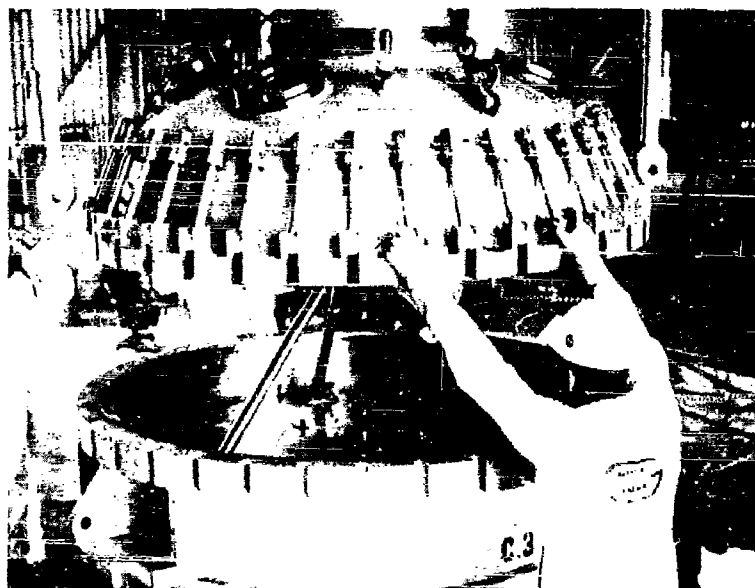


Figure 37 - Placing lid on propellant pot  
and pressure plate in pot

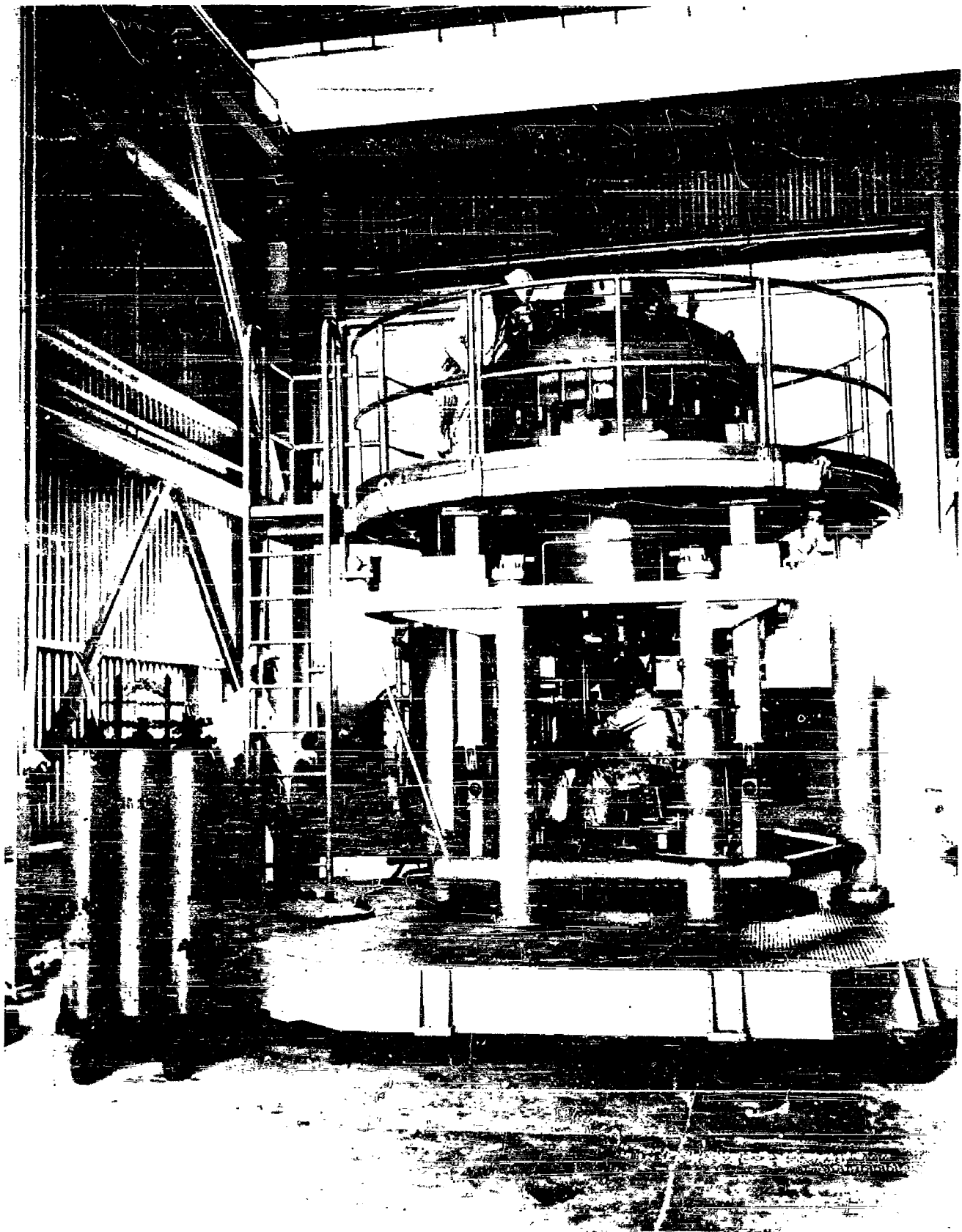


Figure 38 - Propellant pot on casting stand

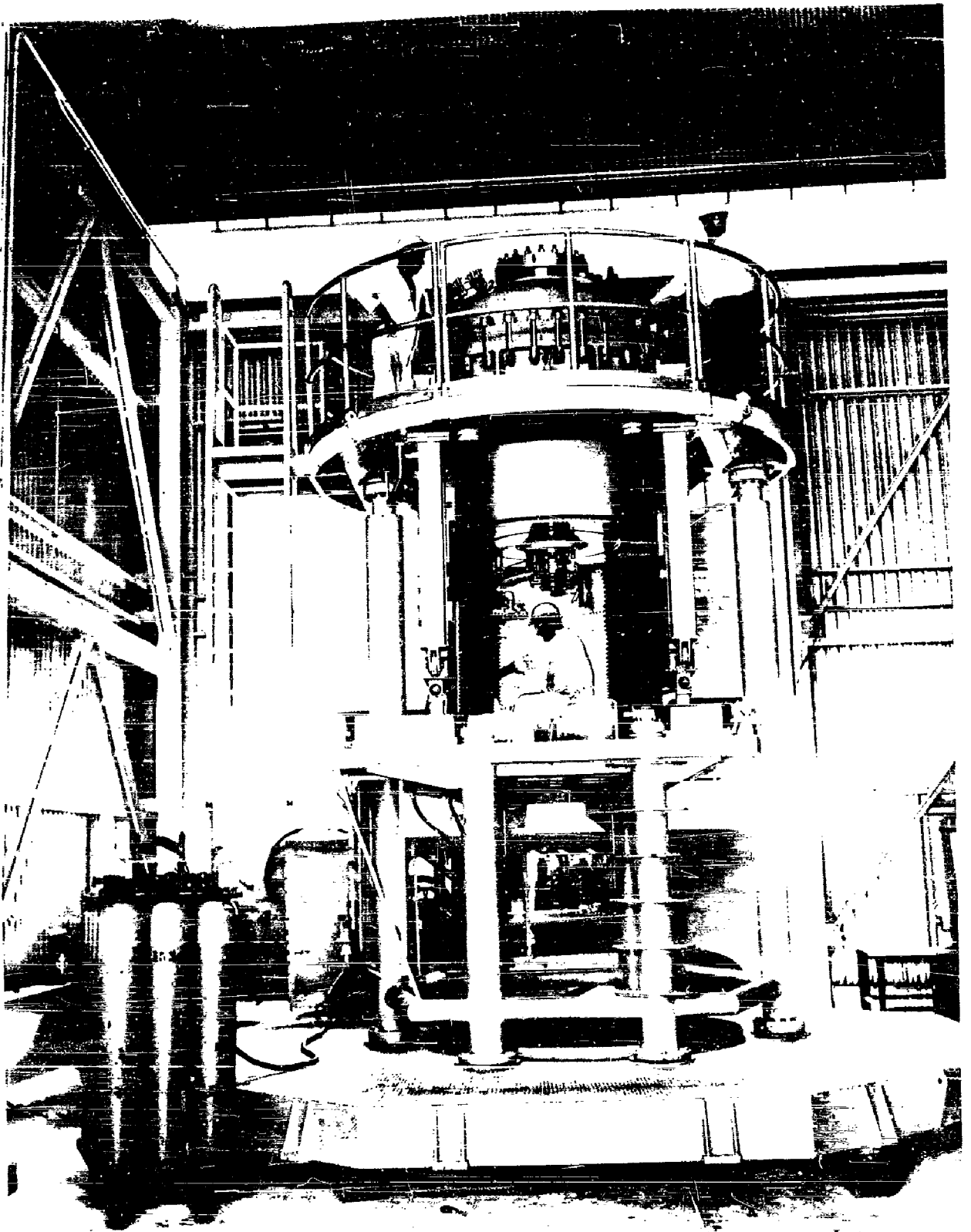


Figure 30 - Propellant pot on casting stand in raised position for bayonet removal

## AEROJET DAMAGE CONTROL SYSTEMS

B. W. Morris  
Aerojet-General Corp.  
Downey, Calif.

### Continuous-Mix Facility

The Aerojet-developed damage-control system is designed to limit the damage caused by a propellant fire to a small part of a solid-propellant continuous-mix facility. Previous experience has shown that a propellant fire within the piping of a plant cannot be controlled by externally applied water; therefore, devices must be installed that are capable of limiting the progress of fire within propellant-filled lines. Valves capable of interrupting a propellant fire have been developed for use in such a damage control system. These valves are controlled by thermostats that are attached to the propellant lines at strategic locations within the plant. The valves are electrically actuated by current supplied from a battery power pack, which is independent from the normal plant power.

In June 1959, Aerojet conducted a study program to establish the feasibility of a system to control the damage within a continuous-mix facility. Results of this exploratory investigation indicated that commercial valves and other mechanisms have not been developed for controlling a fire in a viscous fuel containing its own oxidizer. In a confined volume, a propellant fire will produce gas pressure, which in turn increases the burning rate until an explosion occurs (explosions of this nature are most likely to occur in pumps or mixers). The fire that follows such an explosion could completely destroy a continuous-mix, solid-propellant plant if adequate isolation and extinguishing devices are not used.

A propellant fire differs from a fire involving common flammable materials in that it cannot be extinguished by either smothering or the removal of external sources of oxygen because the propellant contains its own oxidizer (ammonium perchlorate), which supplies oxygen to support combustion. To extinguish a propellant fire, it is necessary to provide a means of conducting heat rapidly away from the burning material, thus cooling it to below its auto-ignition temperature. Water will extinguish a propellant fire if it is applied directly into the propellant line. Heat absorbed in converting water into steam rapidly cools the burning propellant to below its auto-ignition temperature.



As the pressure and temperature to which propellant is subjected increase, its burning rate also increases. For this reason, a local hot spot may ignite confined propellant and cause an explosion without advance warning. The control of a propellant fire, therefore, must be accomplished by the application of two processes: (1) relief of the pressure within the system, and (2) cooling the propellant below its auto-ignition temperature. The burning of propellant within piping may also be limited by inserting a barrier, such as a gate or shutter, which will stop the progression of a fire.

Before any fire protection system could be designed, basic information pertaining to the detonability and burning characteristic of the uncured propellant had to be established. To obtain this information, Aerojet conducted a test program using live uncured propellant made with the same formula that would be produced in the new Dade County Facility.

The first concern in the design of a damage-control system is the sensitivity of the propellant to shock, such as occurs when there is an explosion.

A series of tests was conducted to study the detonability of the uncured propellant slurry under the environmental conditions that prevail within the processing plant. The test procedure consisted of embedding a 1-in. diameter x 1-in. long explosive pellet into a 32-in. length of 8-in. steel tubing filled with uncured propellant slurry. The explosive pellet was electrically detonated; the results, recorded by high-speed motion picture film, showed no evidence that the detonation of the explosive pellet propagated through the propellant-filled pipe. Where a greater degree of confinement occurred, however, the propellant contributed toward the intensity of the explosion.

If explosives were to be incorporated in the design of the damage control system components, it had to be established that their detonation would not start additional propellant fires.

A second series of tests was conducted by detonating small explosive charges within 1/8 in. of the uncured propellant. In several of the tests, a glass barrier was positioned between the explosive charge and the propellant; in other tests, the glass barrier was removed. The results of all tests in this series were identical, i.e., the propellant scattered, but there were no fires.

It is possible that large amounts of uncured propellant might be spilled on the floor of the propellant-processing plant. In the event this propellant should start to burn, it must be known how rapidly a fire would progress; therefore, the following series of tests were conducted:

Approximately 80 lb of uncured propellant was spread in a 12-ft-long strand on the ground, and a motion-picture camera was used to record the progress of the fire along the strand. A clock, positioned in the view of the camera, measured the time required for the fire to burn past wooden stakes placed at 1-ft intervals. Burning rates for ambient temperature and preheated uncured propellant were recorded in this manner.

Propellant-burning rates for 8-in. stainless-steel (open end) tubing were required to determine how rapidly the propellant could be expected to burn within the processing line. In this test series, thermocouples were attached to the interior and exterior surfaces of the tubing at specified intervals. The tubing was placed on end and filled with uncured propellant. After ignition of the top end of the propellant, temperature data with reference to time were recorded on a strip recorder.

As shown by the test results, physical surroundings have a significant effect upon the rate at which a propellant fire will progress. The damage control system must take advantage of early detection to keep the burning rate of the propellant slow enough to permit sufficient time for the control mechanisms to be actuated.

After the basic information pertaining to the detonability and burning characteristics of the uncured propellant was obtained, the next step was to design the fire-protection system. The design of fire control mechanisms for a continuous mix facility presents unique problems. There must be absolute assurance that this mechanism will not allow water to enter the propellant lines during normal propellant processing. There must be no obstructions, ridges, or sharp bends where propellant may accumulate and harden. Therefore, conventional valves, such as those used in chemical plants to control the passage of liquids, are not suitable for use in this application. Another requirement is that the control devices must act much more rapidly than conventional equipment.

To overcome these problems, several types of mechanisms were designed for controlling a solid-propellant fire. Figure 1 illustrates the damage control system that was designed, tested, manufactured, and installed at Aerojet's Dade County Plant.

# EXPLOSIVELY ACTUATED DAMAGE CONTROL SYSTEM

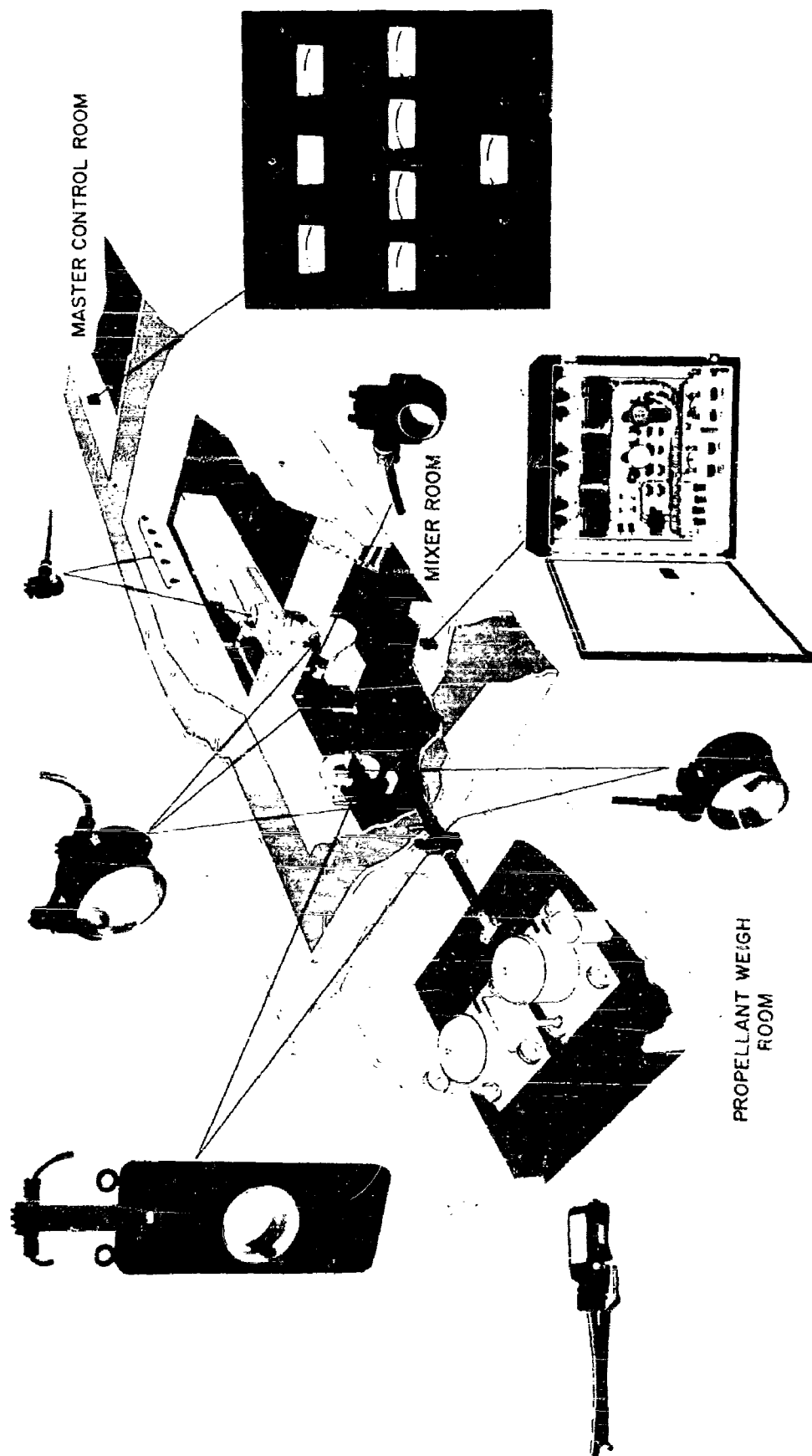


Figure 1. Explosively Actuated Damage Control System.

Electric-sensitive detonators are used to actuate the control valves. These detonators, which are initiated by the discharge of the firing capacitors, may be automatically fired by thermostats or manually fired from a switch in the master control room.

When an explosion takes place or where electrical service power to the mixing plant is off for any reason, a damage-control system depending upon outside electrical power would be inoperative. It was therefore decided to use individual battery power to activate the damage-control system. The power supply is housed within a weatherproof box and consists of dry cell batteries, capacitors, relays, and associated electrical equipment (Figure 2).

Thermostat assemblies are strategically located at potential hot spots throughout the continuous mix facility. Should a propellant fire be started, the thermostats are set to automatically actuate at 250° F, thereby minimizing the delay time in sensing a fire. The actuation of any thermostat will trigger all components of the damage-control system to provide maximum protection in less than 1 second.

When a propellant fire burns within a confined area, such as the propellant piping, pressure rapidly builds up until released by an explosion. To prevent this condition from developing, pressure-relief mechanisms must be placed at strategic locations within the continuous-mix plant.

A fire within propellant-filled piping can be restricted by displacing the propellant with water. The water not only blocks the progress of the fire, but also cools the propellant to suppress the burning.

Deluge valves designed to inject water into a normally closed propellant piping are shown in Figure 3. The relatively inexpensive, reliable, and extremely fast-acting deluge valve has a frangible disc in which small quantities of explosives are embedded. This valve prevents the flow of water at the valve until such time as the system senses a propellant fire; when the signal is received, the explosive detonates and the frangible disc shatters, allowing the water to purge the propellant lines.

To provide a means of isolating one area from another, gate valves (Figure 4) are employed to provide a barrier that will retard a propellant fire.

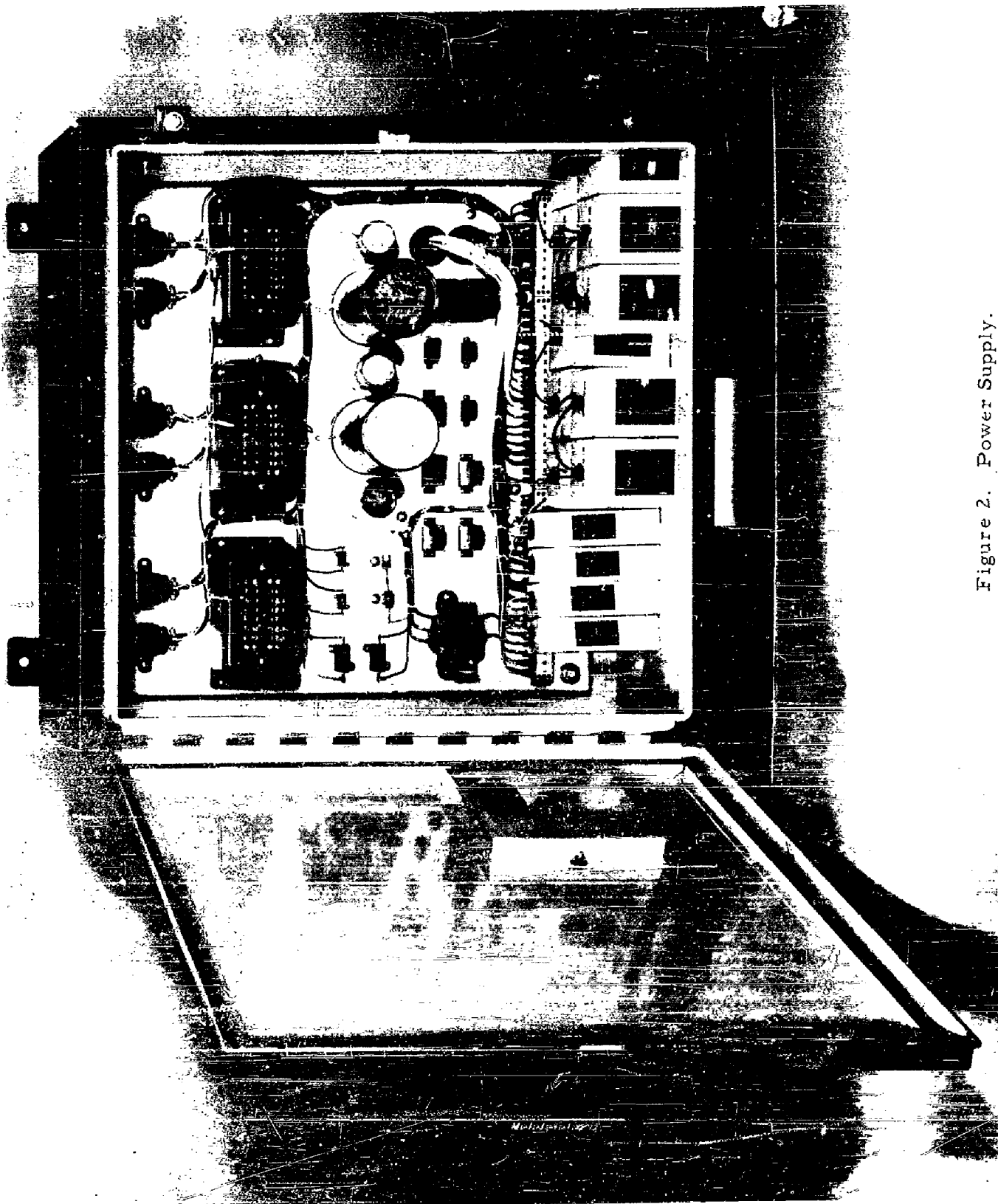


Figure 2. Power Supply.

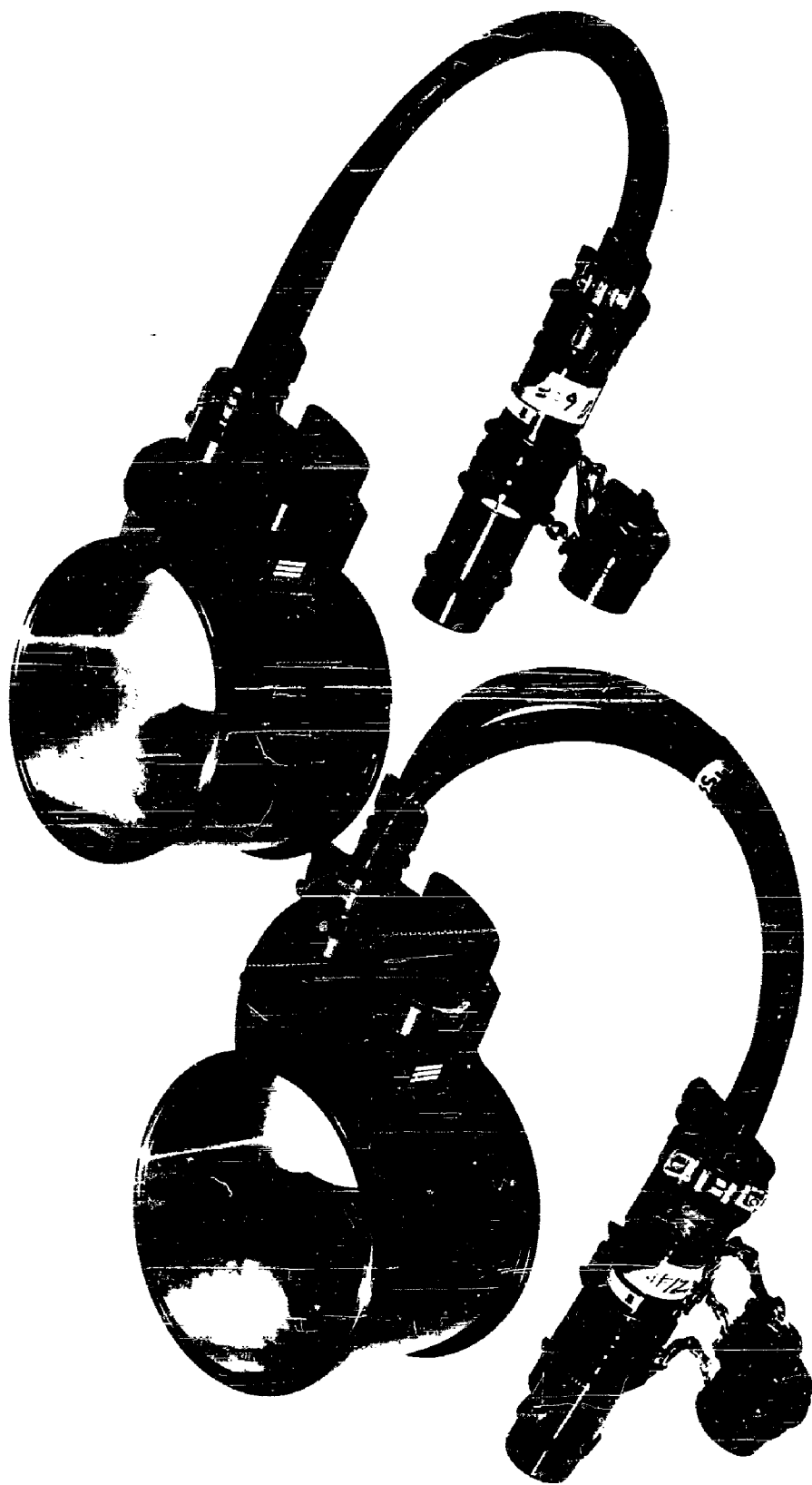


Figure 3. Deluge Valves.

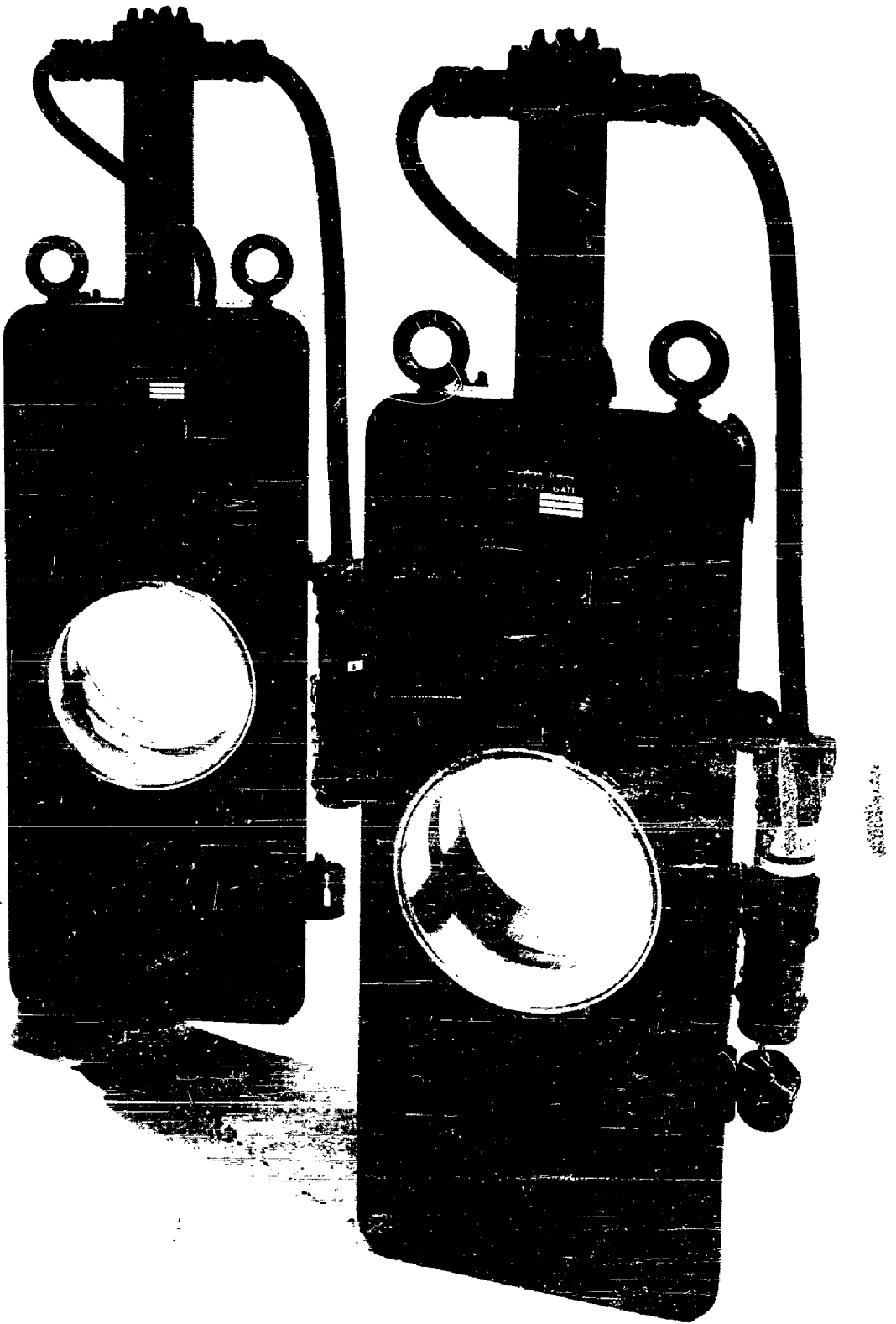


Figure 4. Gate Valves.

A series of frangible disc valves, nozzles, and injectors have been developed to retain water under pressure until activated by an electrical pulse. The design of these valves is similar to that used for the deluge valves, but this is where the similarity ends; the use of the frangible disc valves, nozzles, and injectors is so universal that these items can be applied to any situation where a pipe coupling can be employed.

Some of the design features are as follows:

- a. Size - small and compact
- b. Weight - extremely light
- c. Actuation - electrically initiated or percussion-actuated
- d. Actuation time - less than 10 ms
- e. Burst pressure - in excess of 8000 PSIG
- f. Cost - less expensive to manufacture than conventional valves
- g. Installation - threaded into conventional piping system

Photographs of typical frangible disc valves and nozzles are shown in Figures 5 and 6. The devices are simple, lightweight mechanisms that rapidly open a piping system to allow unrestricted flow. The basic components of the devices are: (1) metal housing, (2) frangible disc, and (3) explosive wire initiator. Opening of the devices is achieved by detonating the explosives, an effect that shatters the frangible disc and permits an unrestricted flow through the valve area.

Aerofit's damage-control system incorporates an automatic electrical checkout system with interlocks to prevent the operation of the propellant-processing equipment if the damage-control system is not in an operating condition. Continuous monitoring of the condition of batteries, firing circuits, and detonators is provided in the damage-control water panel. This panel is located within the master control room of the propellant-processing plant so that checkout and maintenance can be performed without personnel being exposed to the hazards of the mix room equipment.



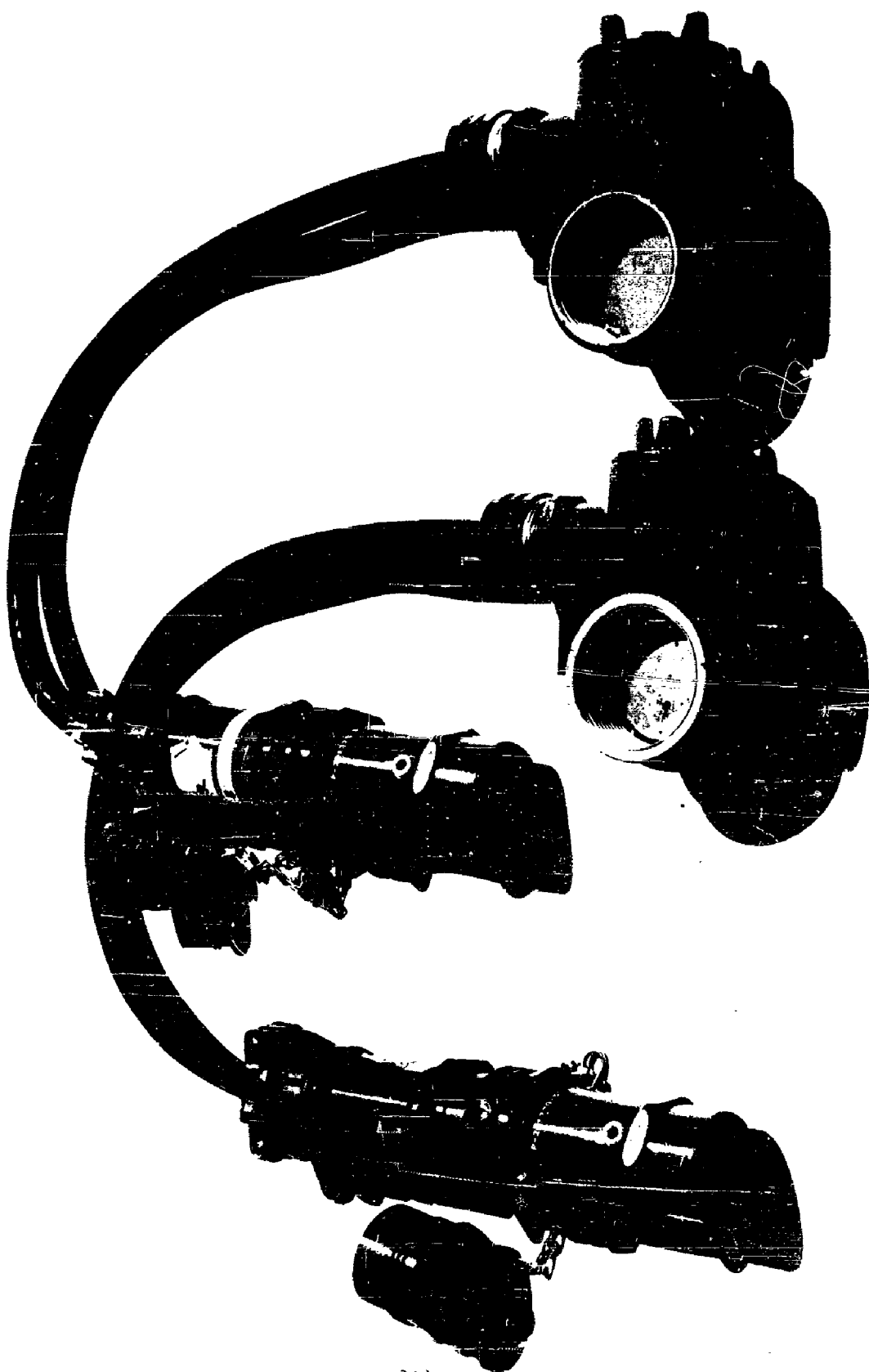


Figure 5. Frangible Disc Valves.

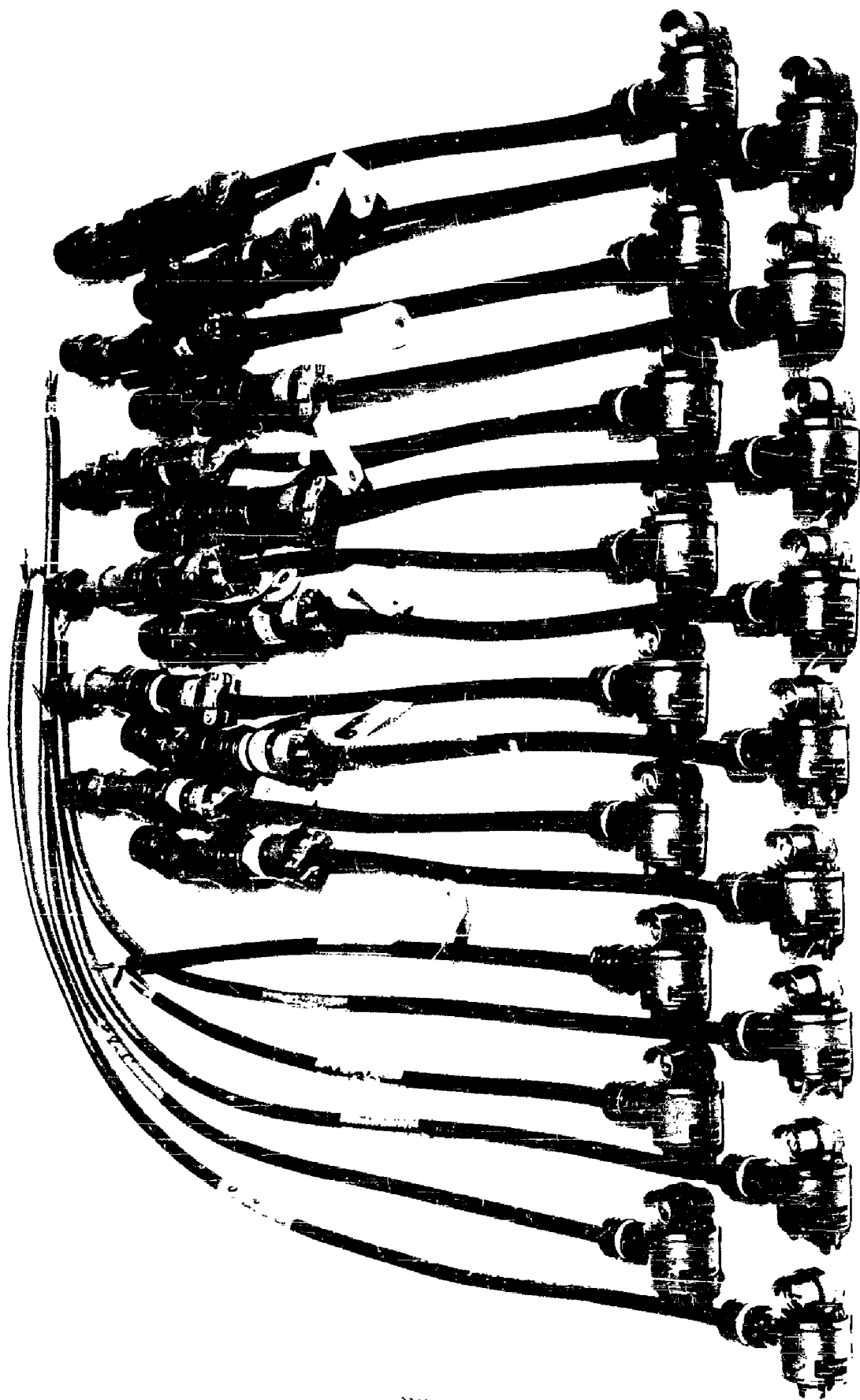


Figure 6. Frangible Disc Nozzles.

### Batch-Mix Facility

An Aerojet-Viking damage control system is installed within two 300-gallon vertical batch mix stations at the Aerojet Dade County Plant at Homestead, Florida. The purpose of this system is to detect a propellant fire and to actuate mechanisms to extinguish the fire. An uncontrolled propellant fire could propagate rapidly and destroy the entire batch-mix building by fire or explosion.

The damage control system performs three primary functions: detection, actuation and extinguishment. Generally, the system will detect a nonstandard condition, such as pressure build-up or fire, in the mixer bowl and actuate mechanisms to relieve pressure and/or allow fluids to spray into the mixer to extinguish the fire.

Detection of a nonstandard condition within the mixer is accomplished by one of two methods: light detection (infrared) or static pressure detection. Each detection system includes detectors, a control unit, and a power supply. The detectors sense a fire or pressure build-up and in less than 35 microseconds send an electrical signal to the control unit, which actuates mechanisms, using battery-supplied current from the power supply, to extinguish the fire or relieve the pressure build-up.

In previous work with propellant fires, it was found that water is an excellent extinguishing fluid. Other extinguishers could be used; however, none are so readily available, reliable, and as inexpensive as water.

After selecting the extinguisher, the type of actuation system must be determined. Many valves, gates, and other mechanical means of introducing water into a mixer are available. All of these "conventional" methods have one serious drawback, however, they require long actuation time. This same problem has also been confronted in previous work with similar systems. It was solved by developing explosively actuated frangible (breakable) disc valves and nozzles, which open in only 7 milliseconds. To complete a fast-reacting system, water must always be present at the valves.

If a propellant fire should be initiated within the batch mixer bowl, the vacuum condition existing during the mixing process would be replaced by an overpressure condition which would increase with time. If this overpressure should be allowed to increase unchecked, an explosion would undoubtedly occur. To prevent this pressure build-up and resultant explosion, a pressure relief mechanism was included as part of the damage control system.

Since the time lapse between propellant initiation and mixer bowl explosion is unknown, it follows that any method of relieving overpressure should be fast acting. The sensing of overpressure within the mixing bowl initiates a frangible disc valve in the bowl's hydraulic system. Relief of hydraulic system pressure allows the bowl to separate from the mixer head, thereby relieving internal pressure.

In addition to protecting the propellant mixing equipment, the damage control system is electrically interlocked to actuate the overhead sprinklers within the batch-mix building.

The damage control system electrical circuits are tested by applying current to the test lead of each detector and, in turn, by receiving a current pulse from the control unit. The circuits for the frangible disc components are tested by measuring the resistance of each detonator bridgewire with a low-energy ohmmeter circuit tester.

Dr. Dale: You mentioned a frangible disc on a branch line on the propellant line to inject water into this propellant line. Is this correct?

Mr. Morris: Yes.

Dr. Dale: Did you use a frangible disc or explosive operated disc on this?

Mr. Morris: It is a frangible disc; however, it is included in the deluge mounting.

Dr. Dale: I'd like to go back to something you said earlier. You had a propellant line and then I gathered you had a branch line in which you had water under pressure ready to be pushed under pressure into this propellant line. This is not a deluge system is it?

Mr. Morris: No, this is the general design. We have water under a head of pressure of 100 psi which is retained by that valve on one side and propellant is being pumped through on the opposite side.

Dr. Dale: How much advance warning did you need to detect this explosion, how much distance or time was sufficient to extinguish this propellant explosion?

Mr. Morris: We didn't have an explosion as such, as you recall in the first film there was no explosion taking place within the propellant. We have a fire generated from some source which is unknown and the propagation of the explosion has not occurred throughout the system and the propellant does not propagate the explosion in this formulation.

Dr. Dale: Have you tried anything that did explode, any formulation that gave you a detonation?

Mr. Morris: The design of our system is for this formulation only. And it was specifically designed for that application, therefore, there was no explosion, we couldn't possibly get an explosion under those conditions.

Dr. Dale: Have you tried any detonation traps, have you worked on any of those?

Mr. Morris: No, we found that it was unnecessary. As I said before in this particular formulation, we could not get it to propagate a detonation. We were very fortunate I think.

Dr. Dale: And on these frangible discs that were explosively operated, can you give me details on those?

Mr. Morris: Yes, there are a number of frangible discs that are available for almost any application. We have been using fixed couplings, pipe couplings, to put the frangible disc in.

## ROCKET MOTOR CASE RECLAMATION

R. A. Yeakey - H. F. McQueen  
Thiokol Chemical Corp., Brigham City, Utah

Hydraulic techniques for removing solid propellant from rocket motors provides a safe and economical means of recovering motor cases. The system capabilities and versatility will be discussed together with the solution to safety problems inherent to hydraulic removal of high energy solid propellants.

Solid propellant rocket motors are designed to have a high mass fraction, that is, to contain as many pounds of propellant in as light a container as possible. The container must serve as a propellant storage chamber, combustion chamber, and often as the air frame of a guided missile. These requirements result in the use of materials with tensile strengths up to 300,000 psi for case fabrication. For large solid propellant rocket motors, these cases range in cost from \$50,000 to \$250,000.

Solid propellants usually consist of a polymeric fuel binder, curing agents, high energy additives, and an oxidizer. The propellant is mixed and cast in the rocket motor case around a core, cured or vulcanized in-place, and bonded to the case wall. After cure, the core is removed, appropriate finishing and assembly operations are performed and the finished motor is shipped to a missile assembly location. Occasionally, casting defects are detected during fabrication that render the motor unsuitable for use, or the rocket motor has exceeded the predicted shelf life, either of these criteria poses a problem to the motor manufacturer

and he is faced with an expensive dilemma.

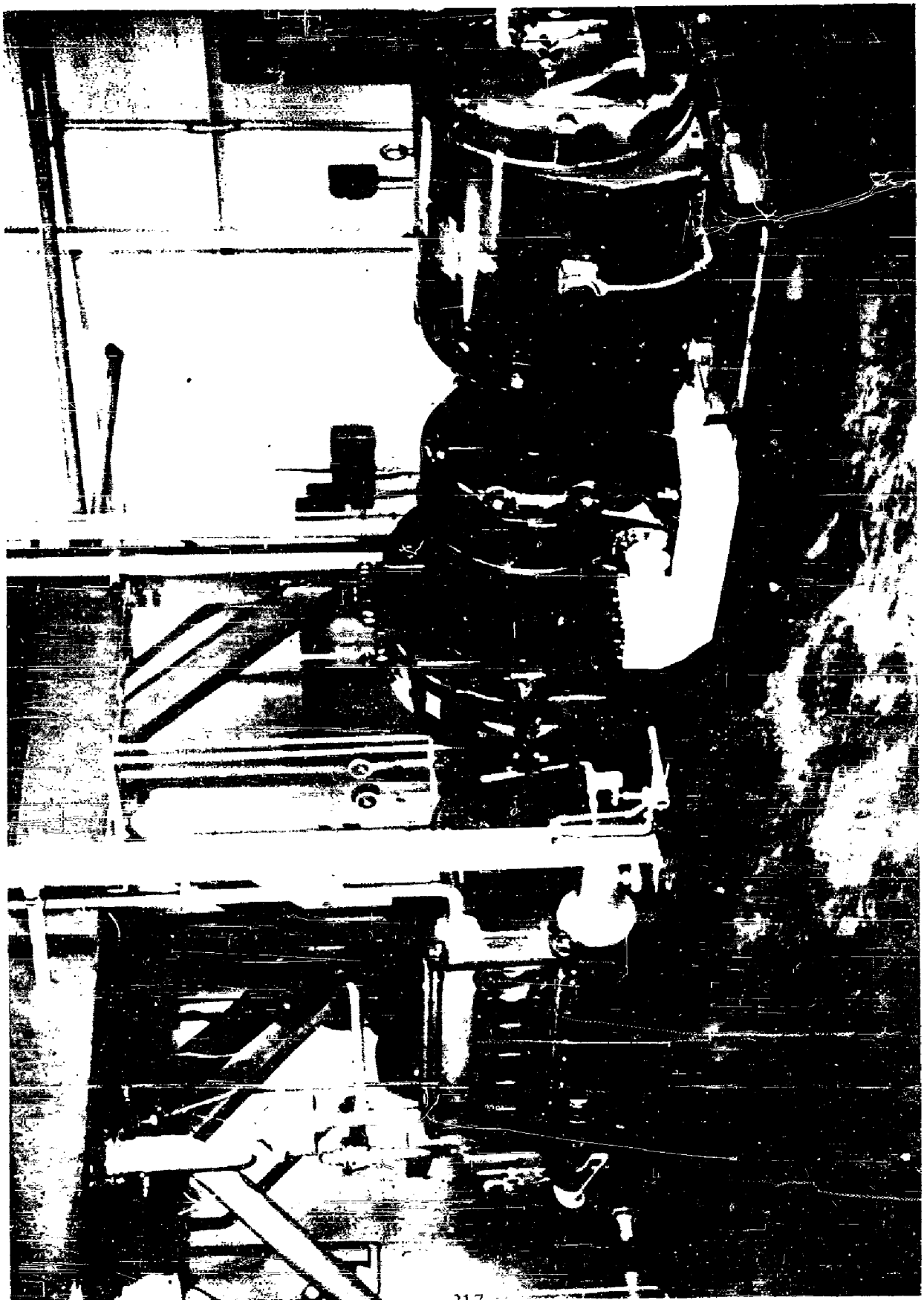
The case is loaded with an extremely energetic, elastomeric, propellant that is relatively friction sensitive and usually resistant to solvents. Conventional burning out of the propellant will destroy the heat treatment and dimensional stability of the case, if it indeed does not destroy the case. Only the more expensive chlorinated solvents have a significant penetration rate, and generally will only swell and degrade the propellant, requiring extensive hand labor for removal. More energetic chemicals usually attack the case.

High pressure water jets have been successfully used to remove sludge, scale, and gum buildup in chemical process equipment. This technique was adapted to solid propellant removal from small, low performance rocket motors as early as 1954. A great deal of development work and improvements have been made since the initial concept. We feel, that at Thiokol Chemical Corporation we currently have the most efficient, most economical and safest method known of hydraulically removing solid propellants from large size rocket motors.

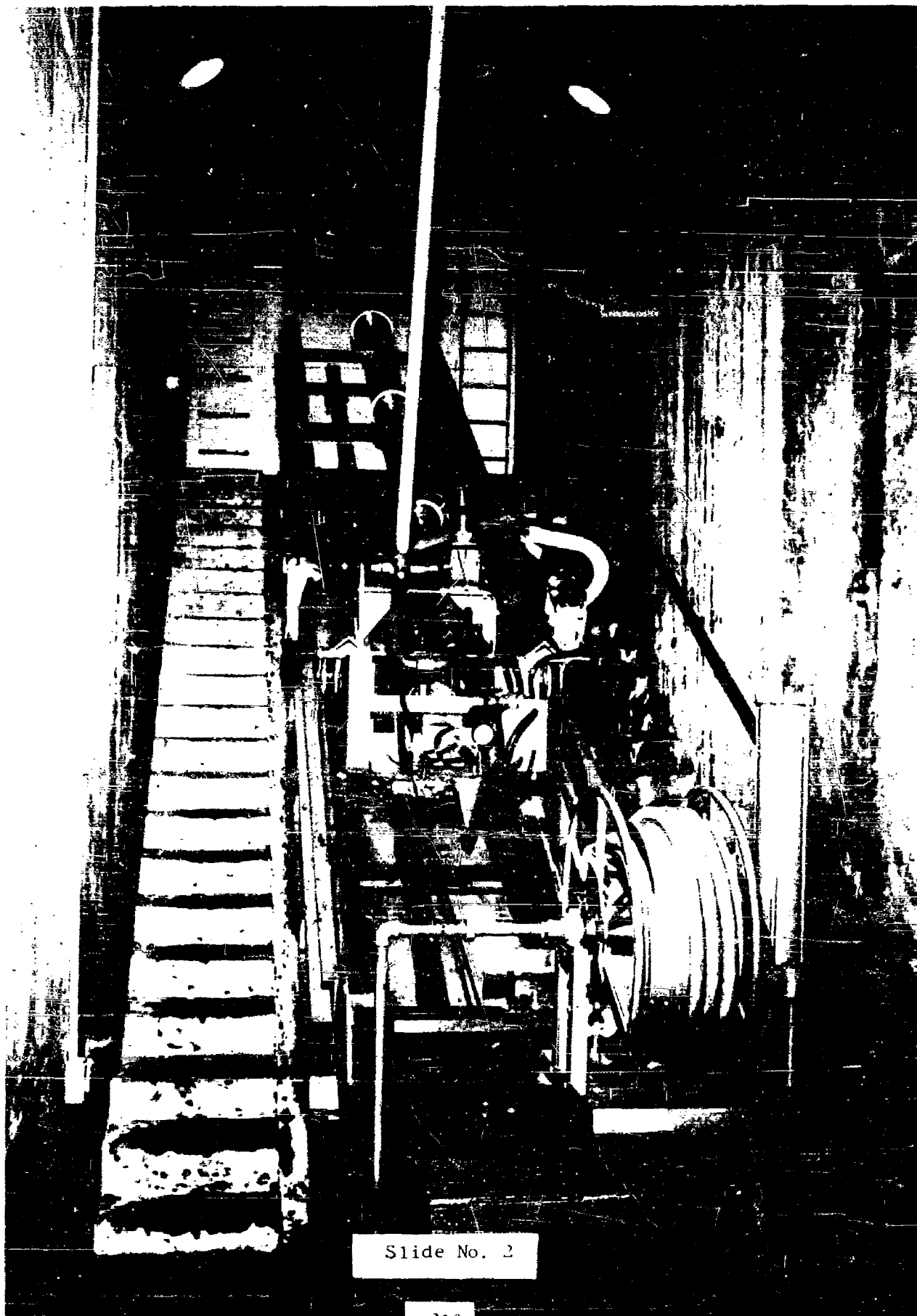
The facility is composed of three systems: a high pressure water system, a de-watering system, and a control system. The water system consists of a 5,000-gallon supply reservoir that supplies a 150 gpm 6,000 psi Frank Wheatly No. 5P 430 high pressure pump (SLIDE 1) and a rotating-retracting boom assembly (SLIDE 2) upon which the nozzle cutting assembly is mounted (SLIDE 3).

(SLIDE 4) This slide depicts a cut-away view of the entire reclamation facility, excluding the remote control room. All blue



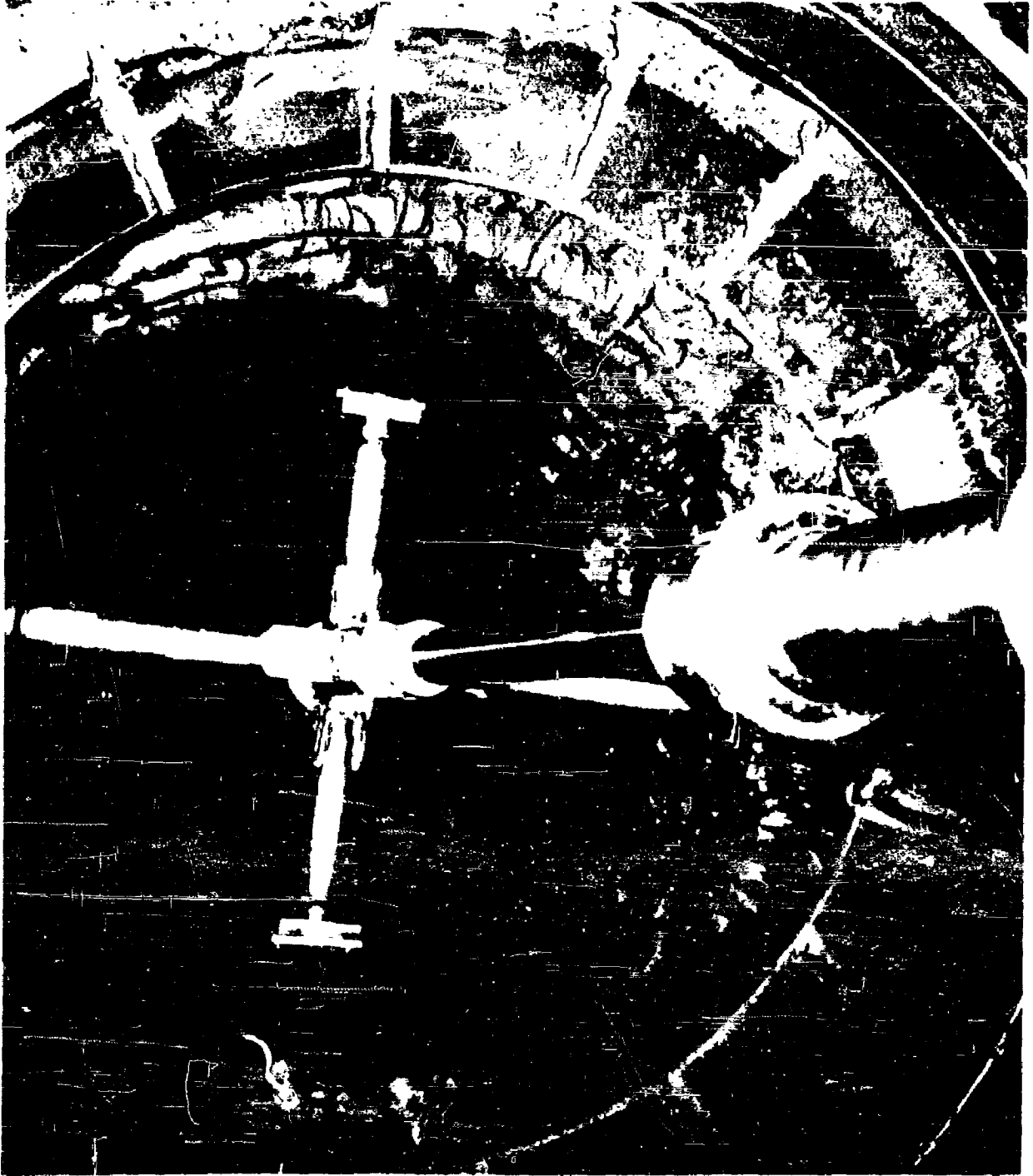


SLIDE No. 1

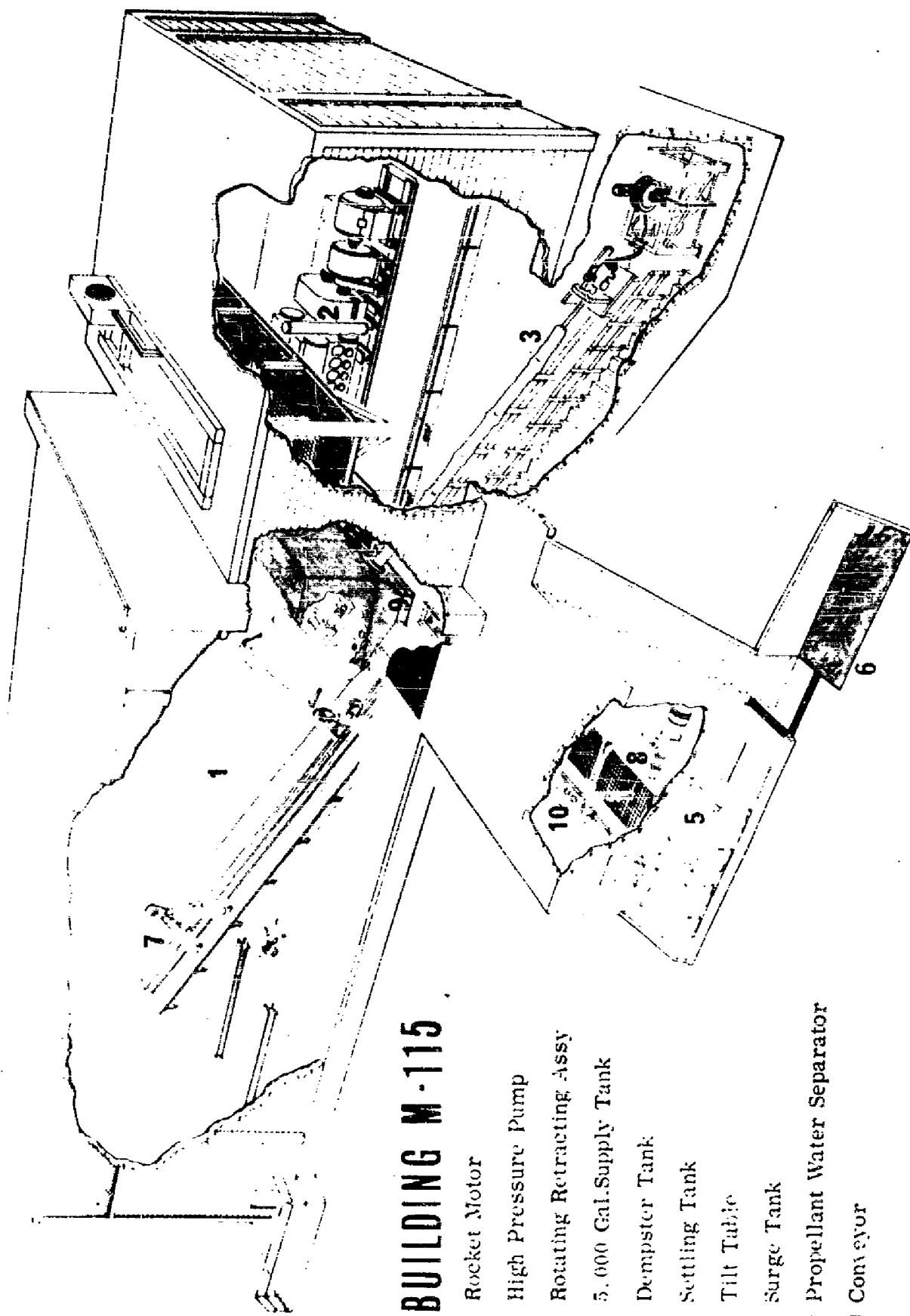


Slide No. 2

NOZZLE  
ASSEMBLY



Slide No. 3



## BUILDING M-115

- 1 Rocket Motor
- 2 High Pressure Pump
- 3 Rotating Retracting Assy
- 4 5,000 Gal. Supply Tank
- 5 Dewatering Tank
- 6 Settling Tank
- 7 Tilt Table
- 8 Surge Tank
- 9 Propellant Water Separator
- 10 Conveyor

colored areas comprise the main portion of the water system. The pump, (#2) receives the water from the overhead tank (#4), the pumping action raises the water pressure to 6,000 psi and is discharged directly to the rotating-retracting boom assembly (#3). From the boom assembly the water is ejected through the cutting nozzles which inturn remove the propellant from the tilted MINUTEMAN motor (#1). The area shown in red is the blast wall which protects the major items of reclamation equipment from the propellant cutting operations. Both the propellant and the spent water fall by gravity to the separator screen (#9). The propellant is conveyed by the oscillating conveyor, (#10) to the portable disposal tanks (#5). The water removed from the propellant by the separator screens flows by gravity to the first of two baffled surge tanks (#6) where the propellant fines are separated from the water. After the settling process is complete the water is pumped back into the 5,000-gallon supply tank which completes the water recycle flow.

SLIDE 5) This next slide is a view of the conveyor discharging the propellant outside the building into the previously mentioned disposal tank. These tanks are conveyed to the waste disposal area where the material is disposed of by burning. (SLIDE 6)

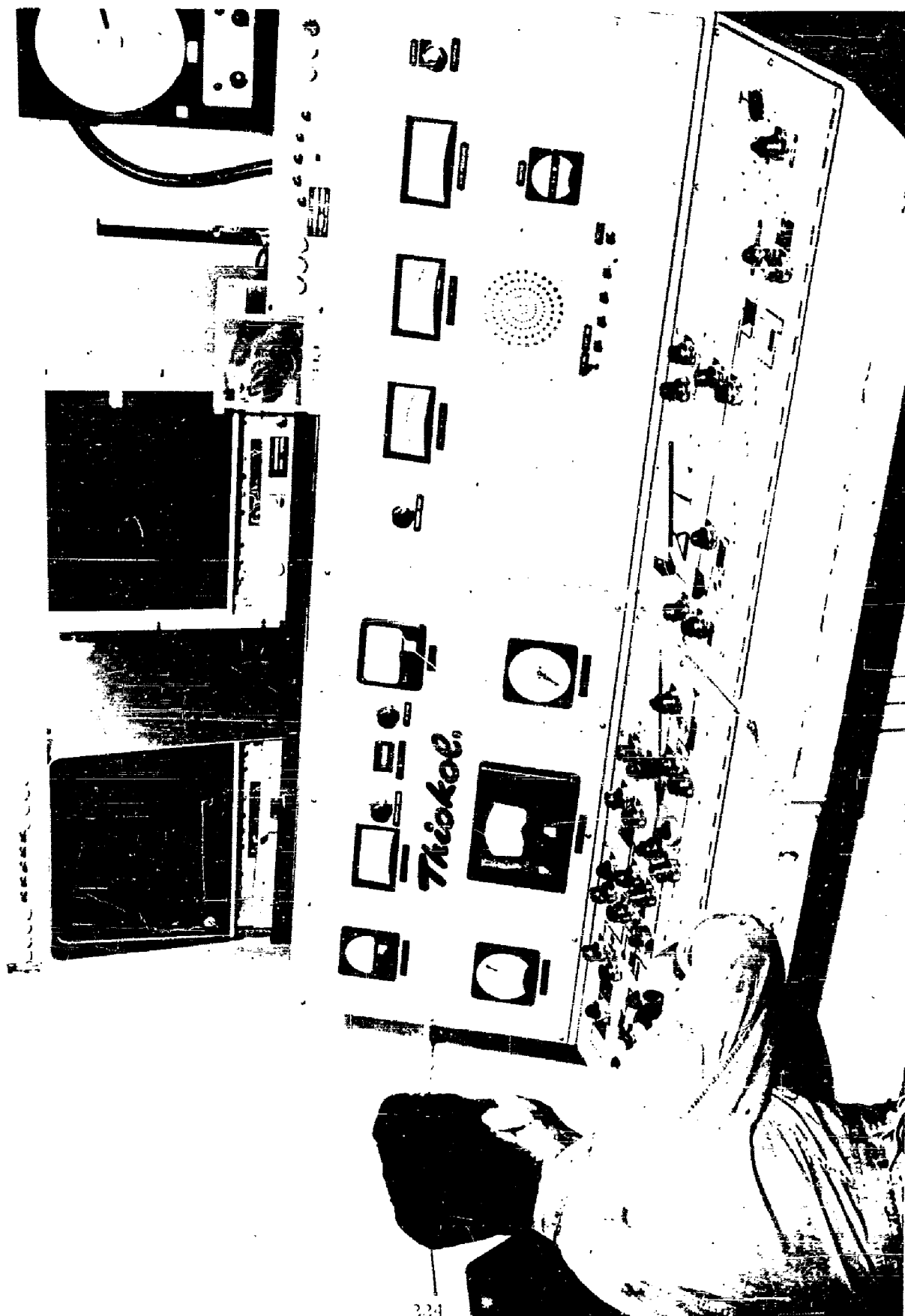
It might be mentioned at this point that, as a general rule, the water soaked waste propellant cannot be successfully ignited using the normal method of "squib" ignition. The propellant must be burned together with normal waste material.

(SLIDE 7) The reclamation facility control system includes local operating stations (for maintenance) and a remote station for

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SLIDE No.7



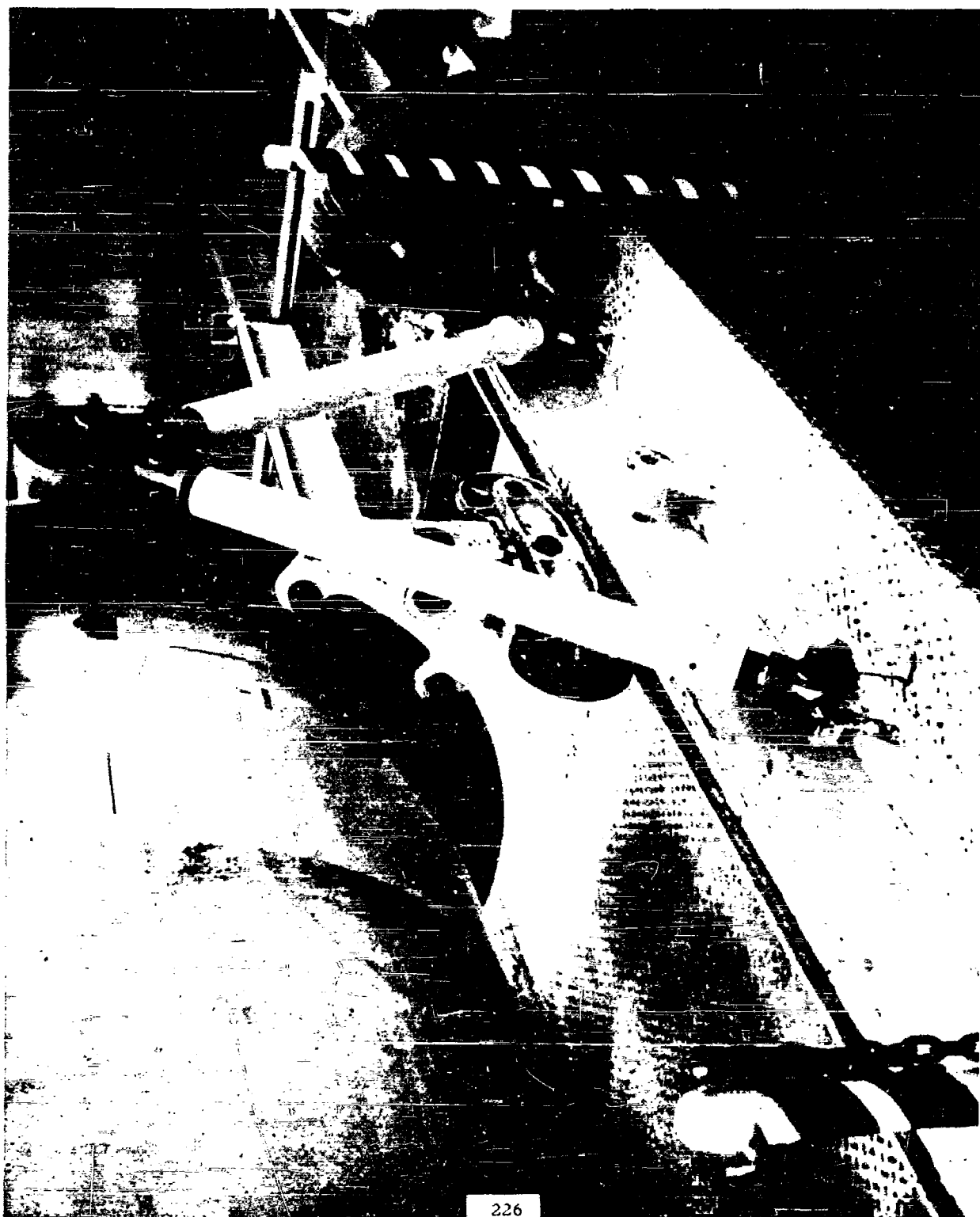
all reclamation operations. The control system consists of a graphic control panel with appropriate interlocks so that a single starting sequence can be used. Failure of any one unit de-energizes all units that precede it in the operating cycle. Closed circuit television is used to monitor the operation.

(SLIDE 8) The motors are positioned on the tilt table with appropriate spray shields and head-end flush water connections installed (SLIDE 9). The motors are then tilted to 30° (optimum) with the aft-end down. (SLIDE 10) This slide shows the same facility being utilized for a Genie rocket motor. The last two slides depict the capability of the facility to process both large and relatively small motors. The MINUTEMAN propellant grain is removed by making several passes with the spray mechanism. (SLIDE 11) Propellant cuts of approximately 6" deep are made with each set of nozzles and extensions.

During the propellant cutout of MINUTEMAN motors, a total of seven nozzle changes are required. This is necessary to maintain the optimum propellant cutting distance of 6 inches.

(SLIDE 12) As can be seen from this diagram, the forward nozzles are positioned to make a lateral cut into the propellant face, while the aft nozzles undercut the propellant in a tangential direction. The propellant chunks fall to the bottom of the tilted motor and are carried to the separator along with the spent cutting water.

Past processing cycle time studies have shown that propellant can be removed from a full-scale First Stage MINUTEMAN motor in 21 hours of pump run time. (SLIDE 13) It is of interest to note





SLIDE No. 9

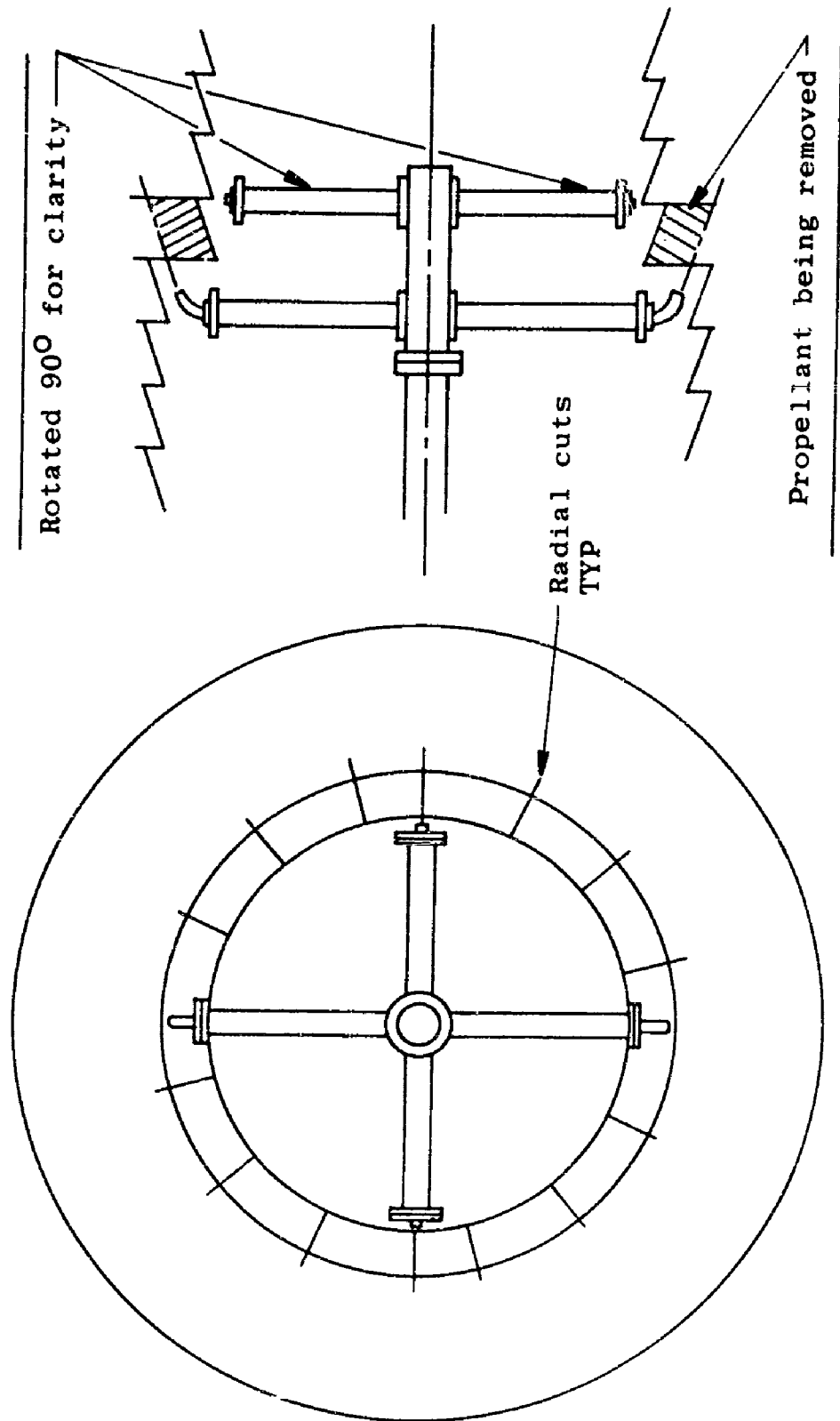


Slide No. 10

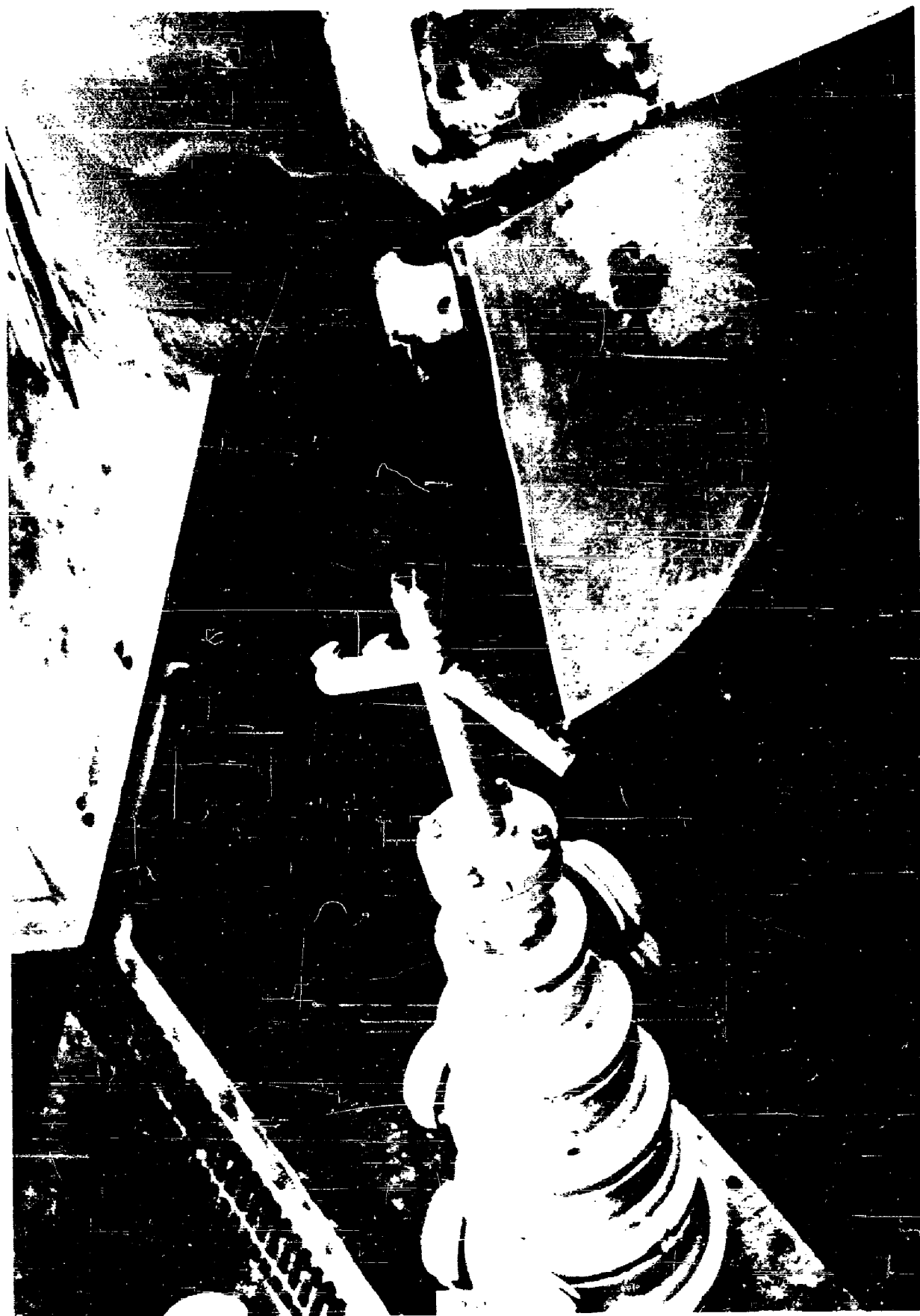


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# CUTTING PATTERN



Slide No 12.



SLIDE 2013

that the Genie motor requires only one nozzle assembly configuration and a single pass of 20-25 minutes duration is sufficient to remove all propellant.

After the propellant, liner and insulation removal operations are complete, the motor is rinsed with hot water to remove the residual salts, air dried, and coated with an interim preservative. (SLIDE 14) Prior to shipment of the case to the vendor, the interior is grit blasted and an (SLIDE 15) ultra-sonic check made of the case wall thickness.

The safety problems encountered with propellant removal are generally the same for all motors regardless of size.

During the initial phases of the facility design concept, it was apparent that two major factors must be considered, these factors were: (1) inadvertent ignition of the motor propellant during cutting operations due to the rapid dissipation of the high energy water stream and (2) continuous operation of a facility in which 6,000 psi water pressure was considered normal operating criteria.

For the purposes of discussion these problems will be considered separately, although there is a certain overlap of the two problems during the actual cutting operations.

The following propellant removal safety criteria were established and the facility was designed accordingly:

1. All propellant removal operations would be controlled from a remote location.
2. All equipment in contact with, or located near, the loaded motor or cured propellant, must be explosion proof.





Slide No. 14



Slide No 16

3. A blast wall would be required between the rocket motor and all other major reclamation equipment.
4. All handling and tie-down equipment must be capable of securing the motor if inadvertent firing occurs.
5. Propellant and process water would be disposed of separately.

The safety requirement that all propellant removal operations would be controlled from a remote location was the prime reason for constructing the nozzle rotating and traversing mechanism. To prevent the boom from contacting the propellant and inflicting damage to the water tube, a key interlock system was incorporated which prevented advancing the nozzle assembly during rotation. Position indicators were required to denote both circular movement and position as well as traverse speed and position.

Because of the relative high velocity impact of the water jet, (1170 feet/sec.) cutting tests were conducted by the Dowell Division of the Dow Chemical Corporation, Tulsa, Oklahoma, to determine if water velocities of this magnitude could induce propellant ignition. Various propellants were subjected to ignition tests. Two of which were: (1) with the pump at 6,000 psi, and using a nozzle diameter of (1/16" to 3/16"), 4" cuts were made without difficulty or ignition, (2) in the second test, a sample of series "E" propellant, procured from the Redstone Division of Thiokol Chemical Corporation was cut without incident. During these tests 5" cuts were made at 6,000 psi with 160° water.

The other major safety aspect to be considered during the facility and tooling concept phase of the case reclamation program was to provide adequate personnel protection from the inherent hazards of utilizing a high pressure fluid as a process media.

Precautions taken to eliminate the possibility of injury to personnel are: (1) The key-interlock system previously mentioned, (2) Operating procedures have been written such that personnel may not enter the propellant removal area without first de-energizing the pump circuit. The key remains in the possession of the person until such time as that individual personally re-energizes the circuit. Because of the high corrosion rate induced by the recycled cutting water, all piping, tanks and the boom are of stainless steel, and (3) a water spray shield is installed over the aft-end of the motor to direct the propellant and spent wash water to the conveying screen. A secondary function of the shield is to serve as a facility protective device in the event of malfunction of the boom; (the shield will contain the water spray and prevent propellant contaminated water from coming in contact with the building equipment.

The film you are about to view depicts the complete case reclamation process and is divided into two parts, large and small motor reclamation. It should be noted that, other than minor tooling changes, the same facility is utilized for both processes. Additional processing steps are required to reclaim the small motors, which necessitated minor facility modifications.

## FILM

Since the start-up of the reclamation facility, on a production basis, there have been no instances of any incidents that could have jeopardized the safety of any of the operating, maintenance or engineering personnel. In spite of this excellent operating record, continuing surveillance is constantly made of the process to determine where processing and safety improvements can be made to the facility.

Naturally Thiokol Chemical Corporation has future plans for continuing and expanding the case reclamation development program. Processes currently under consideration are: (1) Investigate the possibility of hydraulic removal of high energy double-base propellants, (2) Reclamation and reuse of propellant chemicals and (3) Propellant removal from relatively fragile glass wrapped cases by developing more selective methods of propellant cutting.

The initial cost of reclamation facility was \$400,000, and after eighteen months of operation a savings of approximately \$2,250,000 has been realized by reclaiming and refurbishing First Stage MINUTEMAN rocket motor cases.

## STATIC HAZARDS ASSOCIATED WITH PROPELLANTS

Presented at the Sixth Explosives Safety Seminar  
on High Energy Solid Propellants, 18-20 August, 1964

by Ralph L. Mondano, President  
Custom Materials, Inc., Alpha Industrial  
Park, Chelmsford, Massachusetts.

Gentlemen: The evidence indicting static electricity spark as the cause of many explosions and deaths is overwhelming. One of the latest, more serious accidents that falls in this category is the one that occurred at Cape Kennedy on April 14th, causing the premature ignition during spin tests of the X248 third-stage of the Delta Booster. I cite this particular accident because it happened in an area where the humidity is reported to be very high; so high, in fact, that many technical people have seen fit in the past to discount for the most part static spark hazards in such a geographical location.

For the past four years, I have been actively engaged in visiting explosive manufacturing and processing plants throughout the United States and talking to men who have spent many years in the explosive and propellant manufacturing business. This morning I will try to pass along to you the highlights of what I have learned and an understanding of the basic concepts of static electricity hazards. Most important, is how you can reduce this hazard to a point where it is practically eliminated. It would be very presumptuous and vain of me to go into this subject without paying respect to those who have spent several decades researching in the field of static electricity hazards. I have spent a considerable amount of time studying their work.

This presentation reflects the good theoretical and laboratory work by such groups as the Bureau of Mines, Department of Commerce, National Fire Protection Association, Manufacturing Chemists' Association, Hercules Powder Co., Shell Chemical Co., and many others. I couldn't possibly read a complete list, consequently, I have prepared a lengthy bibliography for those who are interested.

About ten years ago, a demonstrated lecture on static electricity was prepared by Mr. William Eathorne and others at the Bureau of Mines. From that date to this, the United States

Government has made this lecture available, for greater safety, to industries requesting it, on a no-charge basis. They have pointed out by visual demonstration the ease with which static sparks can be generated and the ease with which these sparks can cause a fire or explosion. In the original lecture, they discussed briefly such topics as "What is static electricity and where does it come from?"; "How static electricity charges are formed"; and "How static electricity charges may be avoided". I am sure that if the advice given in this last topic heading were followed, several hundreds of lives and billions of dollars would have been saved in the last ten years. I will go into this area in detail in a few minutes.

In January 1959, the Atomic Energy Commission issued a bulletin reiterating the dangers of static electricity sparks and associating these sparks with non-conductive plastics such as polyethylene (copy available). This memo gave actual case histories at AEC sites involving fire and explosions resulting from static charge build-up in the use and handling of finely granulated calcium powder packaged in polyethylene lined drums.

The most recent literature to be printed warning industries in general against the hazards of static spark and, more specifically, the use of non-conductive or polyethylene type plastics in the presence of flammable or explosive materials, is brought out in Volume I of "Case Histories of Accidents in the Chemical Industry", published by the Manufacturing Chemists' Association, Inc. This report points out and describes in detail, with eye witnesses, a number of case histories involving static spark initiated fires and explosions. There is one case in particular, very similar to the AEC case, wherein static spark sensitive powder was being transferred from a drum into a polyethylene bag via the use of a scoop. The operator heard a snap noise like a static spark take place in the bag and the bag burst into flames.

With this background, I would like to go into the meat of my presentation today. Static spark hazards exist where the following four conditions are present:

- I Static charge is being generated.
- II Static charge is being accumulated.
- III A path to ground or a body of different potential is present.
- IV An explosive or flammable substance is present.

As simple and as clear as this appears, time and time again these dangerous conditions exist and are not seen. There are, of course, instances where these factors are present in very complicated arrangements and difficult to recognize, but this is seldom the case. Let us take, for instance, an uncomplicated example (Fig.1). Note the presence of conditions (I, II, III, IV).

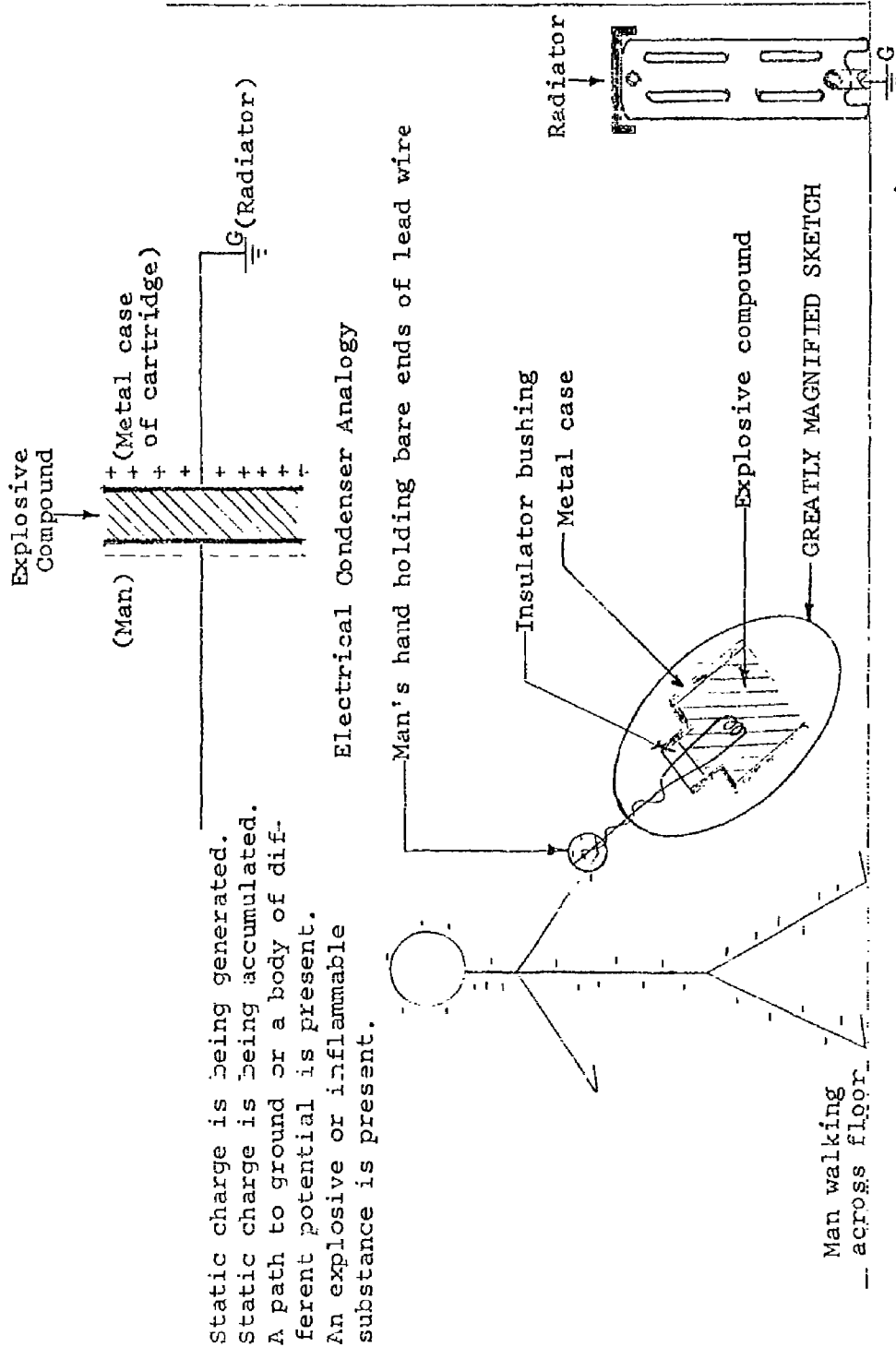
This picture is an actual case history involving an executive in a company that manufactured explosive devices. As you can see in the picture, the man is merely handling the explosive devices in such a way that the explosive compound in the cartridge becomes the dielectric of a condenser. When the executive placed the cartridge device on top of the radiator (shown in the picture) a circuit from the highly charged executive through the condenser to ground was completed. The voltage build-up on the executive was high enough to cause a dielectric breakdown of what in fact was the explosive compound and an explosion resulted. Note the electrical diagram analogy.

Let us now look at another picture involving a missile where-in such a device as this is installed (Fig.2). This picture is considerably more complexed, in that it involves conductive and non-conductive materials which can both act as static charge generators and accumulators. This picture is of a missile sitting on a launching pad under test. Note the squib or cartridge initiating device. Static charge is built up and accumulated, as shown in the picture, and then in a very similar fashion to the cartridge (example shown previously), a spark can occur and prematurely fire the missile.

This, for the most part, according to my unofficial understanding, describes the accident that occurred at the Cape. The majority of people I spoke with at Cape Kennedy are convinced that a static spark jumped from the squib case to the squib lead wires causing the premature ignition of the X248.

It is important for me to point out here that the lead wires could have been grounded or not grounded. A spark could still jump from the case to the resistance element tied to the lead wires, because the lead wires and resistance element were most likely standing at a lower potential and consequently there was an electric stress for an avalanche of electrons to take place. If the wires were grounded, it would just mean that they stood at an even lower potential and an even greater electric stress would have been present.





STATIC SPARK DETONATION  
OF EXPLOSIVE CARTRIDGE  
(electrically actuated type)

Good electrical ground

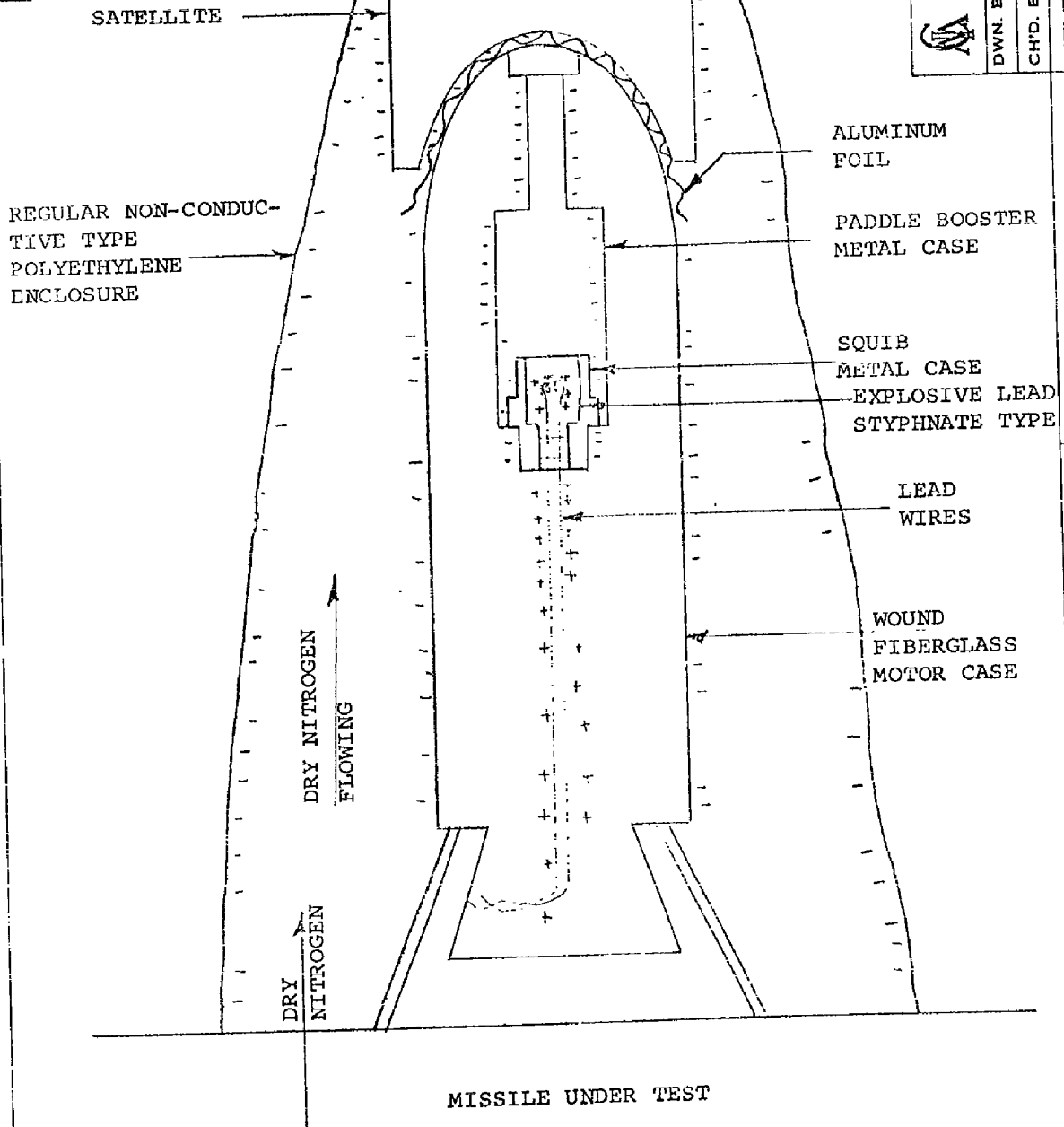
CUSTOM MATERIALS, INC.

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FIG. 1

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DWG. NO. FIG. 2



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I can direct you to many similar case histories involving the handling of flammable and explosive chemicals where similar conditions existed and accidents resulted.

Earlier in my talk, I said that I would refer to a brief list of recommended procedures to eliminate hazards as set forth by the Bureau of Mines. This list is as follows. I have added some points which I have put in parenthesis. These are either for clarification or additions that might be of interest to you.

1. "Elimination of nonconductors as much as possible.
2. Belt drives should be provided with grounded combs or special belt dressings.
3. Dust moving in blower or vacuum systems should have all metal (or conductive plastic) parts bonded (pipe lengths, storage bins, vehicles, etc.) and an effective common ground.
4. Bonding (means interconnecting electrically) of containers or tanks when transferring flammable liquids and also establishing an effective common ground.

(I have written a paper on the "Safe Handling of Waste Explosives and Propellants" as concerns static hazards, and would be most pleased to forward a copy to anyone interested).

5. Movable equipment, such as aircraft, gasoline trucks, etc., should be grounded when they come to a stop to drain off accumulated charges.
6. Preventing liquid drip when transferring flammable liquids.
7. Preventing the escape of compressed air, gases, vapors, and steam.
8. Humidification of the atmosphere to provide a conductive film of moisture on the surfaces of static generating mediums. A relative humidity of 70 per cent has been recommended in recent years, but that may not prevent a charge potential from developing that could set off an explosion.

I have exploded gasoline when the equipment used to generate static (a piece of aluminum and a piece of glass) had been exposed to a relative humidity of 80 per cent for over 24 hours. The only assistance given to remove moisture from the surfaces of the aluminum and glass was wiping them with a piece of cloth that had not been especially dried.

(I didn't want to include this in the list as I feel humidity should never be relied on and should be viewed as a trap. The author, Mr. Eathorne, saw fit, however, to point out the limited value of this technique).

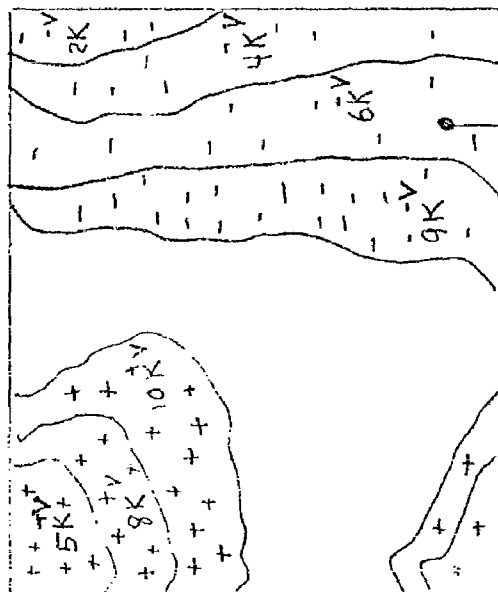
9. Static detecting devices merit consideration where unusual sensitive conditions exist".

Note the item that leads the list is "Elimination of non-conductors as much as possible". Also included in this list is the concept of grounding out dangerous static charge build-up. This is a very important concept because if electrically conductive materials are used and they are properly grounded via secondary grounding systems, no static spark hazard can exist.

The entrance of electrically conductive plastics into the field of propellant and explosive manufacturing and handling has made a significant contribution to safety.

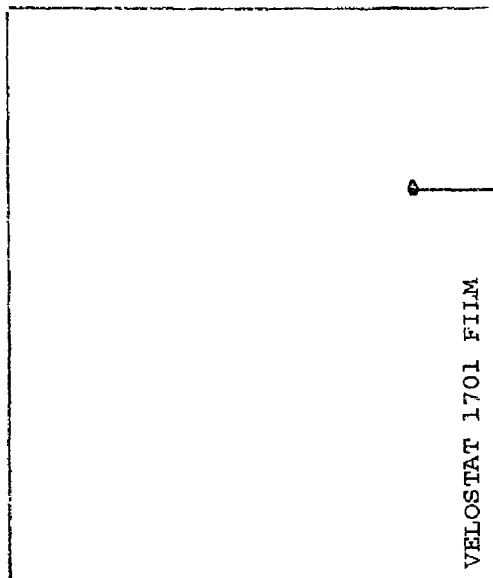
The next picture (Fig.3) shows how electrically conductive plastic performs as opposed to non-conductive plastics. Note you can have erratic static charge build-up on non-conductive plastics and, most important of all, there is no practical way you can ground out this static build-up. The sample opposite it is electrically conductive plastic. By just connecting any spot on this plastic film to ground, you drain off any static charge that is deposited on this type plastic. In fact, conductive plastic film tends to bleed off static charge right into the atmosphere and even when it is ungrounded, it is very difficult to measure any static build-up on conductive plastics. Today, with the help of this new material it is possible to obtain conductive plastic bags, pipe, tubing, gloves, tarps, shrouds, containers, shoe covers, and many more items that are necessary to completely set-up an operational procedure that is free of static hazards. The next picture (Fig.4) shows an example before and after the use of electrically conductive plastics.

NON-CONDUCTIVE PLASTIC FILM




NOTE HOW STATIC CHARGE  
COLLECTS AT RANDOM. GROUND  
DOES NOT REMOVE STATIC  
CHARGE.

CONDUCTIVE PLASTIC



NO STATIC CHARGE CAN COLLECT ON CONDUCTIVE  
PLASTIC. STATIC CHARGE DRAINS TO GROUND  
IMMEDIATELY ON BEING DEPOSITED ON THE  
SURFACE OF THIS TYPE FILM.

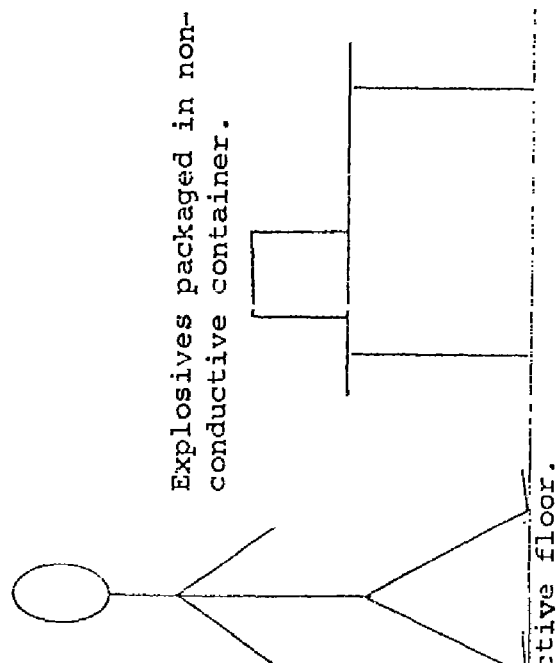
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# EXAMPLE BEFORE THE USE OF ELECTRICALLY CONDUCTIVE PLASTICS

This man is operating under unsafe working conditions as concerns static spark hazard. The package can be heavily loaded with stored static build-up\*, as is the case with foamed plastics and non-conductive plastic film. A spark can occur between the man and the package and pre-detonate any explosive device.



Man not wearing conductive shoe device and table top not conductive.

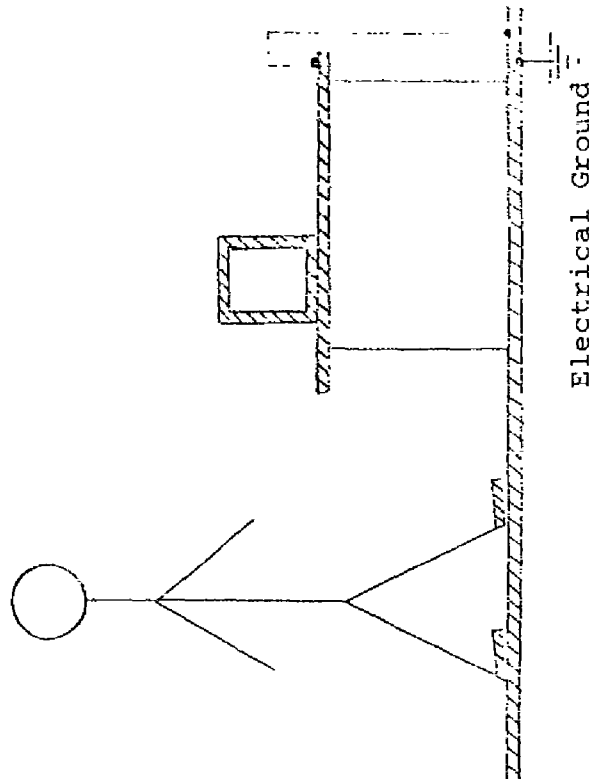
\* Dangerous static charges can be stored over a week (Reference lecture on static electricity by Bureau of Mines).

DWG. NO. FIG. 4

# EXAMPLE AFTER THE USE OF ELECTRICALLY CONDUCTIVE PLASTICS

All surfaces, packaging material, as well as the man in this diagram are grounded and interconnected. There is no potential difference between any of the items and all items are standing in a 100% static-free state.

Designates conductivity



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I would like to make an important point at this time in my presentation, and that is, that again and again I have had manufacturing people tell me that a certain powder or ingredient required for a propellant mix is not flammable or explosive, so why should they request it to be shipped to them in electrically conductive plastic bags. This is a mistake. In my many visits to manufacturing areas I have seen production people pick up one of these static generating bags filled with one of these non-dangerous ingredients, go over to a mixing kettle and empty it into a mix of flammable and explosive solvents and powders.

The Manufacturing Chemists' Association have described several case histories of static spark initiated fires and explosions caused in exactly this way. I would like to caution that you allow no ingredients or materials to come into your plants packaged in non-conductive containers or bags that will be associated in any way with explosives, mixing and handling. This is the exact equivalent of allowing matches and cigarette lighters into explosive areas. The static generating plastic in this regard is even worse because you have no way of protecting yourself from its ever present hidden danger.

The one question that looms greatest in people's minds concerned with the subject of static hazards, is "When do I know if I have a static hazard problem?" There are on the market today, static meters. These are very easily operated and can indicate the presence of static electricity charge build-up. The following picture shows a typical static measuring gun (Fig.5). There is no electrical measuring device that can take into consideration all of the variables associated with static electricity build-up and flash a red light when the danger level has been reached. Therefore, the meter performs its function in informing you that static build-up is present. After this, the procedure is simply that you must employ conductive materials and grounding procedures to eliminate this static spark hazard.

I would like to caution you against the use of so-called static-free plastics. These materials are not electrically conductive and cannot function as secondary grounding systems and, consequently, cannot drain static charge build-up to ground. So-called static-free materials are for use in areas such as paper handling, woven fabrics, etc. and are not recommended for use in the environments with which we are concerned.

The next picture (Fig.6) shows a chart of dangerous energy levels for a number of flammable or explosive materials which we readily recognize. I have also put on this chart, at the bottom, the level of energy which a person can easily build up on his body during many routine work tasks, which is .015 joules. This number

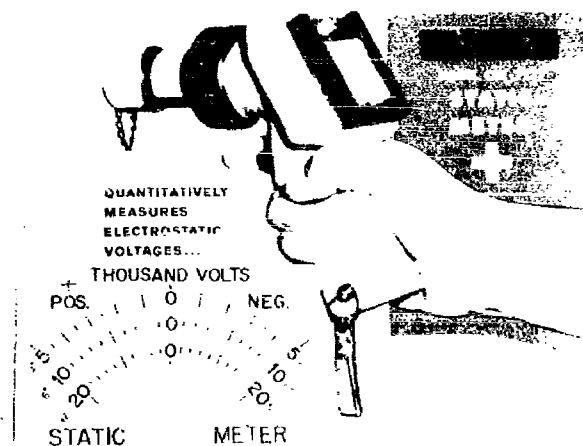


Figure 5 - Typical static meter designed in pistol fashion

The static meter shown has three scales of 0 to 5,000 volts, 0 to 10,000 volts, 0 to 20,000 volts. It then has an orifice device that can be used to provide a multiple of ten times the scales indicated. In other words, 0 to 50,000 volts, 0 to 100,000 volts, 0 to 200,000 volts. This meter can be used to determine the presence and level of static build-up.



FIG. 6

IGNITION CHARACTERISTICS OF SOME SOLVENT VAPORS  
AND EXPLOSIVES

SELECTED MINIMUM

VAPOR	IGNITION TEMPERATURE		MINIMUM IGNITION ENERGY IN JOULES
	IN AIR °F	IN OXYGEN °F	
Acetone	1042	905	.0006
Benzene	1076	1051	.0005 - .001
Carbon disulphide	248	225	.00015- .0002
Diethyl ether	379	367	.00045- .0015
Ethyl alcohol	738	-----	.00065
Gasoline	536	-----	.001
Heptane	446	417	.0011
Hexane N	478	-----	.00072
Methyl alcohol	878	862	.0005

	MINIMUM IGNITION ENERGY IN JOULES		MINIMUM IGNITION ENERGY IN JOULES
A/KC/04 Igniter Pellets	0.250	Potassium Chlorate-	
HMX SR-28-61 TYPE A	0.250	Lead Sulfoeyanate	
Nitro Cellulose Type A	.075	Mixture	<.00075
Nitro Cellulose (13.4% N)	.062	TNT (through 100-mesh)	.062
Resorcinal	.075	Tetryl granular (through	
HMX SR-27-61 Type B	.05	100-mesh)	.007
Zirconium Powder	.00125	PETN (through 100-mesh)	.062
Aluminum Powder	.625	Ammonium picrate	
Ammonium Perchlorate	>6.25	(through 100-mesh)	.025
Casting Solvent 70% NG	>6.25	Nitrostarch	.007
Casting Solvent 90% NG	>6.25	Igniter composition	.21
Lead Azide	.007	Mg powder, grade B	.007 to >12.5
Mercury Fulminate	.025		
80/20 Mercury Fulminate Potassium			
Chlorate Mix	.054		
Lead Styphnate	.0009		
DDNP	.012		
Tetracene	.010		

$$C = \frac{KA_{10}^{-3}}{d} \quad E_{\text{joules}} = 5 \times 10^{-7} CV^2 \quad \begin{matrix} (V - \text{volts}) \\ (C - \mu\text{f}) \end{matrix}$$

$$Q = CV \quad E_{\text{joules}} = \frac{1}{2} CV^2 = \frac{1}{2} QV = \frac{Q^2}{2C}$$

$$E_{\text{joules}} = VA \text{ Sec.}$$

Human Body Capacitance .0003  $\mu\text{f}$   
(Bureau of Mines)  $E = .015 \text{ joules}$

was determined by the Bureau of Mines. Work completed by others has this closer to .03 joules.

Also on the bottom of this chart are two formulas, one for capacitance build-up and the other for energy build-up so that you might see the relation between energy, capacity and voltage. At this point the formulas are purely academic and as I have already mentioned are unable to be applied simply to obtain specific level of static spark hazards.

Remember that energy build-up is much greater for operations such as sliding non-conductive plastic bags over large configurations or by the flowing of air through duct work or liquids through pipes. Voltages in the order of tens and hundreds of thousands of volts are often times measured in these latter type operations.

In conclusion, please allow me to reiterate the evidence is overwhelming indicting static spark as the cause of many fires and explosions. While we don't completely understand the involved technicalities of the subject, we do have a definite procedure to deal with the problem. Conductive plastics are now available in volume to assist in putting into practice the grounding procedures that will eliminate the hazard. Your field of propellants, liquids, solids and combinations is becoming more and more complex. In many cases, new propellants and explosives are manufactured and used before all their associated hazards are known. By utilizing the proper grounding procedures, you will be taking positive action to eliminate any associated static hazards, known or unknown.

Your help in putting to use the facts I have tried to assemble and present to you today can save many lives and much property. You now have a new tool, electrically conductive plastic - put it to work. The next series of slides will show you how conductive plastics can operate in theory in actual applications.

The next slide (Fig.7) shows a small Van de Graaff generator with strips of non-conductive polyethylene standing out from the charged sphere. Basically, this is law of like charges repelling each other. Before the sphere is charged up, the plastic tentacles will hang limp as shown in Fig.8. The hand in Fig.7 cannot conduct away the static charge build-up because the plastic is non-conductive.

Fig.9 shows the same arrangement utilizing conductive plastics. I could not show a picture of a hand touching one of the tentacles and still have them lift away from the sphere. The sphere in Fig.9 is charged as highly as possible by turning the hand crank and the tentacles are just barely pushed away from the sphere and drop very quickly because of the leakage of the charge from the conductive tentacles into the atmosphere.

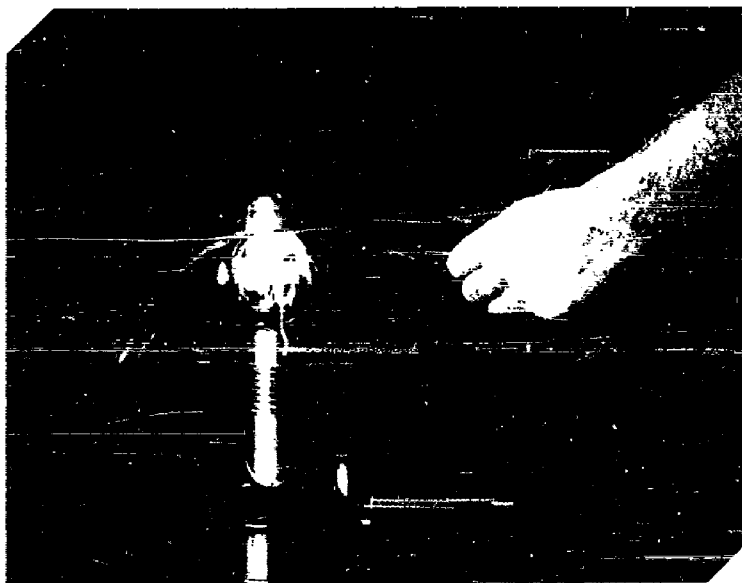


Figure 7

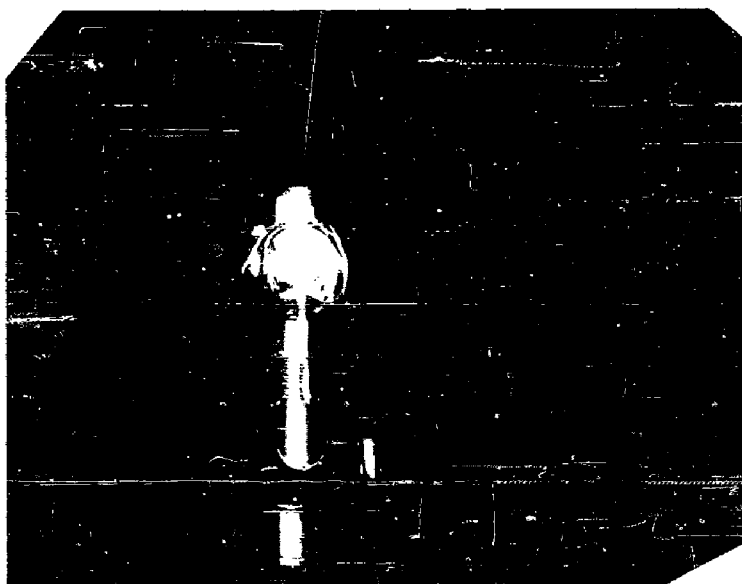


Figure 8

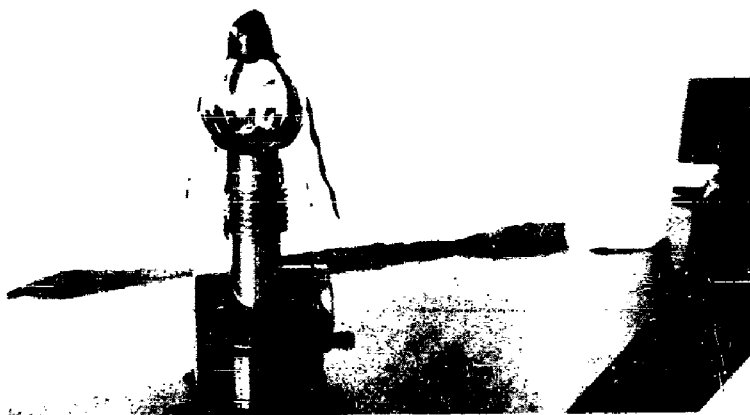


Figure 9

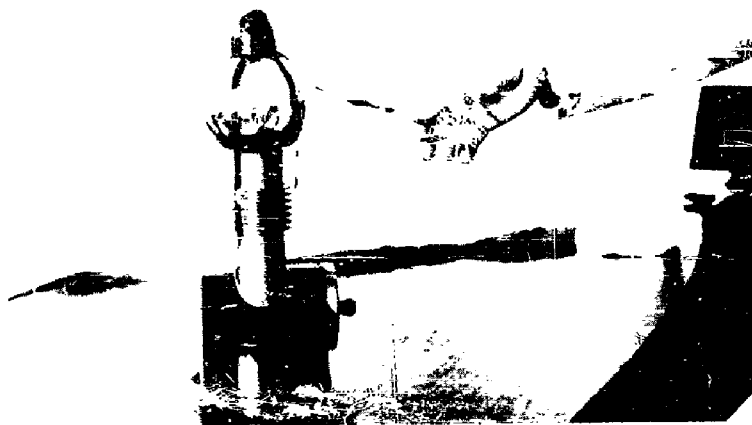


Figure 10

Slide 10 shows what happens when one of these tentacles is touched. Note you immediately drain off all static by flow to the body.

Fig.11 and 12 show a technician standing on a conductive mat, which could be a conductive floor, reaching for a bag that is on a conductive table top. The bag is also electrically conductive. Note in Fig.11, before he touches the bag there is no meter reading which indicates no electrical continuity in the circuit. If you note in Fig.12, when the man touches the bag he closes a loop and the meter shows a reading. Now, if this system is grounded, it provides the maximum protection against static electricity hazards.

I now will demonstrate the point brought out in these last two slides.



Figure 11



Figure 12

# Ref. B                      BIBLIOGRAPHY - LIST OF ARTICLES ON STATIC ELECTRICITY

<u>TITLE:</u>	<u>SUBJECT:</u>	<u>AUTHOR:</u>
Static Electricity	NFPA No. 77M	National Fire Protection Assoc. 60 Battery March St., Boston, Mass.
Standard for Aircraft Fueling on the Ground.	Recommendations - NFPA No. 407	National Fire Protection Assoc. 60 Battery March St., Boston, Mass.
Code for Use of Flammable Anesthetics.	NFPA No. 56 Safe Practice for Hospital Operating Rooms.	National Fire Protection Assoc. 60 Battery March St., Boston, Mass.
Aircraft Fueling Up to Date	Aircraft fueling	Joseph M. Chase, Flight Safety Foundation, 468 Park Avenue S., New York, N.Y.
Manual on Static Electricity in Aircraft Operations and Main- tenance.	NFPA No. 404M	National Fire Protection Assoc. 60 Battery March St., Boston, Mass.
"Dusts, Fumes, and Mists in Industry".	A National Safety Council Technical Service	National Safety News
"Annual Report of Research and Technologic Work on Explosives, Explosions, and Flames. Fiscal Year 1946".	Report of Investigations	Bernard Lewis, Bureau of Mines
"Examination and Treatment of Industrial Magnesium Foundry Wastes".	Report of Investigations	O.C. Garst, Bureau of Mines

Ref. B (Continued)

TITLE:

SUBJECT:

AUTHOR:

"Annual Report of Research and Technologic Work on Coal".	Information Circular	A.C. Fieldner and R.E. Brewer, Bureau of Mines
Case Histories of Accidents Caused by Static Sparks	Case histories	National Fire Protection Assoc. 60 Batterymarch St., Boston, Mass.
"Electrostatic Ills and Cures of Aircraft"	Electrification of Airplanes	Robin Beach, Fellow A I E E Brooklyn, N.Y.
	Letter re Static Sensitivity of Black Powder	The National Board of Fire Underwriters
Static Electricity	Cause, Effect, and Elimination	Warren W. Levy, The Simco Co. 920 Walnut St., Lansdale, Pa.
"Radio Frequency Energy"	A Potential Hazard in the Use and Transportation of Electric Blasting Caps	Institute of Makers of Explosives 250 E. 43rd St., New York, N.Y.
Explosives	Rules for Storing, Transporting and Shipping	Institute of Makers of Explosives 250 E. 43rd St., New York, N.Y.
Fire and Explosion Hazards with Flammable Anesthetic Agents and Their Control	Death by Anesthetic Explosions	George T. Thomas, M.D., University of Pittsburgh School of Medicine, Pittsburgh, Pa.
"Rocket Fuel Bottleneck is Broken by Bulk Bins"	Article on Bulk Bins	Chemical Processing for Operating Management, Texas
"Detecting the Saboteur Static Electricity in Gasoline Fires"	The Violation of Safety Practices in the Handling of Gasoline	Robin Beach, Fellow A I E E Polytechnic Inst. of Brooklyn, New York, N.Y.



Ref. B (Continued)

<u>TITLE:</u>	<u>SUBJECT:</u>	<u>AUTHOR:</u>
"Fire and Explosions Due to Electrostatic Charges in the Plastics Industry"	Fire Prevention Bulletin	SPI Committee
"Lecture and Demonstration on Flame Propagation and Permissible and Explosion-proof Electrical Equipment"	Combustible Vapors and Gases Information Circular #7980	Hal H. Engel and W.M. Merritt Bureau of Mines
"Flammable Materials: A Lecture Demonstration"	Fire and explosion hazards of Flammable Liquids and Solids	M.G. Zabetakis and H.H. Engel Bureau of Mines
"Consultation Corner"	At What Speeds Do Belts Produce Static Electricity?	L.C. Smith, Industrial Dept. NSC
"Chemical Safety - a session with the experts"	Questions and answers on static electricity	Article in "Industrial and Engineering Chemistry"
"Let the Belt Carry the Static"	Static conducting V-Belt	Frank H. Rumble, Texrope Drive Specialist, Allis-Chalmers Mfg. Co., Milwaukee, Wisconsin
"Anti-Static Agents...The Do-It-Yourself Approach"	"How to Make Plastics Static-Free: Part 2"	H. Earl Tremain, Assoc. Editor
"Demonstrated Lecture on Static Electricity"		William Eathorne Bureau of Mines
"Static Electricity Can Kill Transistors:		Donald G. Stroh, B.K. Sweeney Mfg. Co., Denver, Colorado

Ref. B (Continued)

<u>TITLE:</u>	<u>SUBJECT:</u>	<u>AUTHOR:</u>
"The Theory Behind Static Electricity Hazards in Process Plants"	Article from "Chemical Engineering"	D.I. Saletan, Engr.Dept., Shell Chemical Co., New York, N.Y.
"How to Calculate and Combat Static Electricity Hazards in Process Plants"	Article in "Chemical Engineering"	D.I. Saletan, Engr.Dept., Shell Chemical Co., New York, N.Y.
"Bibliography of Bureau of Mines Investigations of Coal and Its Products, 1910-60"	Information Circular 8049	Bureau of Mines
"Keep Static Electricity Static!"	Static Electricity Hazard in Industry	Safety Maintenance
"Electrostatics in the Petroleum Industry"		A. Klinkenberg and J.L. van der Minne
"Recent Studies on the Explosibility of Cornstarch"	Report of Investigations 4725	Irving Hartmann, Austin R. Cooper, and Murray Jacobson, Bureau of Mines
"Bibliography of Bureau of Mines Health and Safety Publications July 1, 1955, through June 30, 1961"	Information Circular 8121	G.G. Morgis Bureau of Mines
Laboratory Equipment and Test Procedures for Evaluating Explosibility of Dusts	Report of Investigations 5624	Henry G. Dorsett Jr., Murray Jacobson, John Nagy, and Roger P. Williams - Bureau of Mines
"Explosibility of Dusts used in the Plastics Industry"	Report of Investigations 5971	Murray Jacobson, John Nagy and Austin R. Cooper - Bureau of Mines

Ref. B (Continued)

<u>TITLE:</u>	<u>SUBJECT:</u>	<u>AUTHOR:</u>
"Static Electricity in Hospital Operating Suites: Direct and Related Hazards and Pertinent Remedies"	Bulletin 520	P.G. Guest, V.W. Sikora, and Bernard Lewis - Bureau of Mines
"Aluminum Paint Hazard Suggests Annotated Code Need"	Thermite-type reaction	Health and Safety Information United States Atomic Energy Commission.
"Static Electricity"	Safety Newsletter	National Safety Council Chicago, Illinois
"Magic of Fire"	Notes to accompany a demonstration lecture	William Eathorne Bureau of Mines
"Static Electricity in Industry"	Grounding Principles and Practice V-static electricity in industry	Robin Beach, Head Robin Beach Engineers Associated Brooklyn, N.Y.
"Safety from Industrial Electro-static hazards"	Control of electrostatic fire and explosion hazards in industry	Robin Beach, Head Robin Beach Engineers Associated Brooklyn, N.Y.
"Static Electricity on Rubber-Tired Vehicles"	Study of the fundamental elements of static electricity on rubber-tired vehicles	Robin Beach, Head Robin Beach Engineers Associated Brooklyn, N.Y.
"Electrostatics in the Rubber Industry"	Control of electrostatic fire and explosion hazards throughout the rubber industry	Robin Beach, Head Robin Beach Engineers Associated Brooklyn, N.Y.
"What Makes a Static Spark?"	Prevention of static dangers	Fire Protection for Industry

Ref. B (Continued)

<u>TITLE:</u>	<u>SUBJECT:</u>	<u>AUTHOR:</u>
"Sensitivity of Explosives to Initiation by Electrostatic Discharges"	Report of Investigations 5002	F.W. Brown, D.J. Kusler, and F.C. Gibson U.S. Dept. of the Interior
"Static Electricity"	General outline of the various ways in which manifestations of static electricity introduce appreciable hazards into many industries.	Francis B. Silsbee Bureau of Mines
Case Histories of Accidents (including fires and explosions)	Case Histories	Manufacturing Chemists' Association
"polyethylene Static Hazard"	Issue No. 88 - Accident and Fire Prevention Information	United States Atomic Energy Commission
"Measurement of Leakage Resistances and Static Electrification"	An investigation performed in The National Vitamin Laboratory, Copenhagen, Denmark.	Bent Barnholdt, M.S. and R.E.H. Rasmussen, Ph.D.
"Facts About Electrostatic Charges"	Electrostatic spark hazards	Safety Newsletter
"Static Electricity in Nature and Industry"	Bulletin 368	Paul G. Guest Bureau of Mines

Mr. Harton: I want to point out that the X-248 investigation has not been completed. I want to also point out that the statements made here were not cleared through NASA. I can't speak for the Air Force. We hope to get out some appropriate information for those who have a legitimate need-to-know. I do not consider it appropriate to request information about NASA accidents from Mr. Ullian. I don't really want to get into this X-248 details today.

Mr. Mandano: Well I would like to state that the information I have put together here is based - you notice I didn't refer specifically to the diagram as being the X-248 - I just drew a mock-up and showed a follow-through of a squib going into this device and what could have happened.

Mr. Harton: The implication was there however.

Mr. Mandano: I'm sorry it was, I didn't intend it but what I wanted to do was to point out that these things can happen. Again, in my paper I pointed out that it was not an official presentation at all, just a presentation based on study and analysis and what could have happened so please don't misconstrue the information.

Mr. Diebold: You had a squib with a metal case around the explosive charge and you said that this is a hazardous condition due to discharge between the case and the propellant?

Mr. Mandano: Yes.

Mr. Diebold: Now as I understand it, with your conductive material you want to first make a sack out of this material and then place our non-conductive propellant inside the sack and then ground the sack or whatever you want to do with it?

Mr. Mandano: When you have a conductive material your conductive material goes over a metal object - say it was touching a conductive box sitting on top of the motor. Now any charge that is collected on that box is immediately grounded through the cover and charge collecting on that plastic would be grounded through the cover. It is an electrical ground.

Mr. Diebold: Going back to the man walking along with the pair of squib wires in his hand and he sets the squib on the radiator. This is the same as far as I can tell as a man walking along with a propellant or explosive in a polyethylene sack that is conductive and he sets it on a grounded table and if his shoes are not properly grounded and if other situations exist, this would be an identical situation I believe.

Mr. Mandano: Analyze that for a minute. The man has a non-conductive bag in his hand, the propellant is inside. Any flow of current would go through the lowest resistance. So, therefore, when he touches the metal radiator the current would flow through the bag to the radiator would it not?

Mr. Diebold: Right.

Mr. Mandano: It would not necessarily fire any sparks in the system to the propellant.

Mr. Diebold: You may have a point there but at this time I cannot see the distinct advantage or the difference between the squib situation and the propellant in the conductive sack. There may be a difference there but its a little obscure to me at the moment. Perhaps I should speak to you later.

Mr. Mandano: I see the difference now, you're referring to why would you put materials in conductive bags. Is that the question?

Mr. Diebold: That is correct.

Mr. Mandano: Many times in the case of powders when you ship them you will build up a static charge due to friction and as a result this charge, if it were not in a conductive package, could not be drained to ground until somebody came close enough or something came close enough for a spark to fire it. This is the same principle as putting a ground wire on a metal container that you store powder in a storage building. You have a source to drain that charge to ground. That is what the conductive bag will do for you, it gives you an opportunity to drain that charge from what's inside, drain that charge from the contents to ground. Maybe we could carry this on during intermission.

Mr. King: I was advised to something that seemed very interesting to us as a result of this unnamed incident. I'd like to throw it out for your consideration. We were advised on good authority that the nitrogen purge in this situation did not contribute anything to the development of a charge. This was contrary to what we had felt in the beginning and we were further advised by several reliable sources that the only thing that the nitrogen would contribute was to the extent that it would reduce the humidity and it would contribute an ionizing effect only to the extent that it had impurities in it. I wonder if you would comment on this and other points.

Mr. Mandano: Well I have been working in this area to determine just what gases are more susceptible to causing or are more apt to cause a lot of static build-up when they pass over surfaces and I have come up with one conclusion contrary to some of the literature - that all gases will create a situation where a charge is dissipated on the surface that it passes over. So I can't answer that part of your question as accurately as I would like. But one area I feel I can answer and that is that because the gas was dry you definitely multiplied your problem of charge in the system.

Mr. Owings: Since we are destined to die with our pneumatic conveying systems, I take some exceptions to statements that you made and would like to have equal time here but since I've already had my say and time is running short I'll talk to Customs Materials later.

Mr. Mandano: John, I understand what you're saying but I hope that everyone here understands that I come in here simply so that we could all learn a little more. I am a student, I'll do my best in the years to come to contribute as much as I can in the static field. Please do not judge me too harshly until I've spent at least ten years in this business. Thank you.

THE DIVIDING WALL TEST PROGRAM DOCUMENTARY FILM

Russel G. Perkins  
Armed Services Explosives Safety Board

Mr. Perkins presented a film entitled "Some Problems in the Storage of Explosive Ordnance" which reviews the background, accomplishments and future plans of a coordinated test on explosives structures which is monitored by the ASESB and jointly funded by the three Military Departments and the Defense Atomic Support Agency. The film is being reviewed by the Defense Department with the intention of producing it in an unclassified form. After this review, if the material can be issued in a completely unclassified version, it is planned that it be made available to appropriate Government film distribution points such as the Navy Photographic Center and the Army Film Library for further distribution at cost to all organizations and individuals having an interest in the technical subject matter presented.

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Mr. King: I think one of the films showed that the density of fragments was greatest near the center of the wall and least near the corners, yet I recall you mentioning that the pressures experienced insofar as you can measure them are greater at the corners. Was the decreased density due to the fact that the corner came out as one piece or have you any comment on that?

Mr. Perkins: I think certainly the break-up of the wall varies between the corners and the centers and depending upon placement of the charge. The pressures also vary depending upon the configuration of the cubicle and the placement of the charge so that you can't really make a general answer to such as this. The break-up of the corners is usually to a smaller number of larger pieces.

Mr. Stosz: My question concerns one of the value or significance of the earth fill between your metal structures. I noticed or it seemed to me that distances between these particular magazines were greater distances between similarly loaded concrete structures. Have you any evidence that the earth actually helps?



Would it be as effective to have an air space there and what would be the relative protection provided by concrete structures as for separated by air?

Mr. Perkins: We don't have a complete answer to such a question as this. The distances approved for separation of steel arches were less than - significantly less than - the standard separation presently approved for concrete arches. The earth fill shown in three of the tests to two feet above the top is of course the maximum which was put in. The fourth test showed a configuration of the earth cover over the magazines approximately comparable to that which is standard for a regular concrete magazine. The Board accepted anything between, but you have to have at least that much earth cover, you can't use the arches without earth cover or with less than a two on one slope, I believe it is.

## SAFE HANDLING OF IMINE CHEMICAL COMPOUNDS

H. F. Krackenberger  
Thiokol Chemical Corp.  
Longhorn Army Ammunition Plant  
Texas

### INTRODUCTION

The use of imine curing agents to extend the low temperature operational limits of castable composite propellants based upon polybutadiene acrylic acid co-polymer and a newly developed carboxyl terminated polybutadiene polymer was recognized as early as 1959, when highly loaded propellants were static tested successfully at temperatures as low as  $-75^{\circ}\text{F}$ . Since that time, continuous development efforts by Thiokol Chemical Corporation and other solid propellant manufacturers have yielded a wide series of new highly dependable propellants, compatible liner, and insulation systems using imine cure agents. At the same time it has been made eminently clear that new techniques are necessary in handling the imine chemicals to guard against not only the toxicological but also the fire and explosive hazard present in the use of these materials. The information presented here is intended to point out areas where caution need be exerted, evidence for the safe use of imines, procedures that are recommended for safe and proper handling, and suggested methods for use in monitoring the concentration of imines in production.

### IMINE CURING AGENTS

Of the wide variety of imine curing agents currently being produced in the industry, only a few have proven satisfactory for use in manufacture of solid propellants. The few selected to date have demonstrated a controlled rate of reaction, suitability for mixing with other propellant raw materials, and capability to produce desirable physical properties in propellants. Probably the most widely recognized imine in use today is MAPO\*. MAPO's chemical name is tris [1-(2-methyl) aziridinyl] phosphine oxide. The chemical formulation is shown in Figure 1. MAPO is a highly reactive tri-functional derivative of phosphorous oxychloride and propylene imine. Other imine cure

\*Interchemical Corporation, Commercial Development Department, New York, N. Y.

(Note: Pertinent references are indicated by parenthetical notations: (Refs. 1,2,3-5). The list of references is at the end of the text, just ahead of the illustrations.)

agents are generally of the form 
$$\text{R}-\text{N} \begin{array}{l} \diagup \text{CH}-\text{CH}_3 \\ | \\ \text{CH}_2 \end{array}$$
 The radical, R, may

be an aliphatic or aromatic structure as well as a triazine, sulfone or nitrogen based aliphatic, Figure 2. Since MAPO has received considerably more attention than other imines and possesses the hazardous characteristics of other imines to varying degrees, we will consider it in detail rather than use generalities of the entire class of imines.

#### PHYSICAL PROPERTIES

Depending on the chemical structure, the imines may vary from solids to liquids. MAPO is a straw colored liquid with a specific gravity only slightly higher than water. During normal storage, no fumes or vapor are visible but a definite amine odor is present. It is completely soluble in water and most organic solvents. As long as it is kept free from moisture or acids, it is stable at normal room temperatures. Other imines may require low temperature storage to assure stability.

#### PHYSIOLOGICAL PROPERTIES

Although the physiological properties of the imines have not been investigated in great detail, sufficient information (Refs. 1-13) has been gained through recognized test methods to indicate that toxic effects of these compounds can be induced by contact in either the liquid or vapor form through absorption, ingestion or eye contact.

Based on the test for dermal toxicity of household products, according to the procedure of MCA (Manufacturing Chemists Association), MAPO is considered to be toxic by absorption through the skin. Tests in which a 30% solution of MAPO was applied to the skin of rabbits resulted in 100% fatalities within three to ten days. By contrast, the ethylene imine analogue, tris (1-aziridinyl) phosphine oxide, produced the death of all test animals within 24 hours. The autopsy results and the symptoms seen in these animals suggested an enzyme inhibition, which allowed an excess of acetylcholine to accumulate (Ref. 2). Excessive levels of acetylcholine can affect nerve endings and thereby contribute to pulmonary edema, fixation of the respiratory muscles or respiratory failure (Ref. 3).

Although the dosage required to kill small animals by skin absorption is above that likely to be encountered by a careful worker, it is evident that contact of MAPO with the skin should be avoided. Technicians

working with imine curing agents have developed transient skin irritations and dermatitis. Prolonged exposure to imines, epoxides, and phenolic derivatives has also induced sensitization to a degree that no further exposure to raw chemical materials could be tolerated.

The oral toxicity of imines presents an equally hazardous problem. As a result of studies in which imines were administered orally to white rats, the "Estimated Median Lethal Dose"--that dose expected to kill half the animals tested--was determined to be 192 mg/kg for MAPO. This would make MAPO comparable to DDT in toxicity through ingestion. Systemic poisoning occurs with death resulting from damage to the lungs, kidneys and other organs.

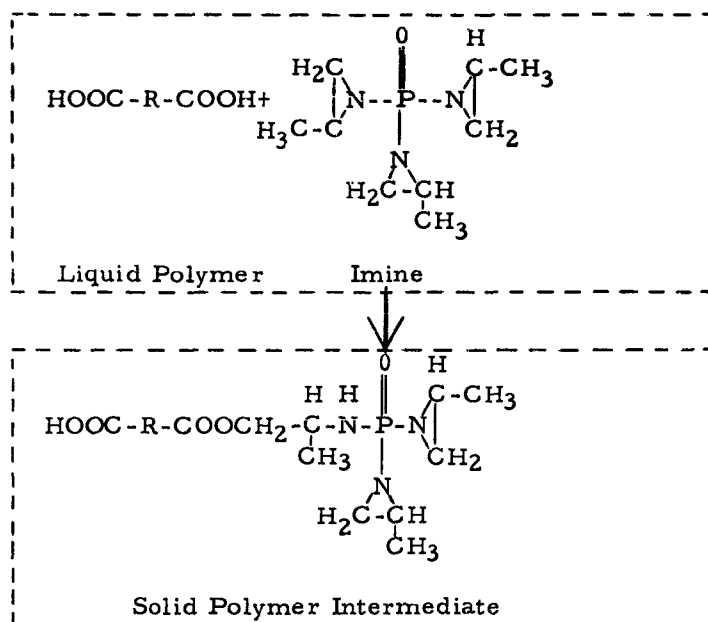
Eye contact is a problem of irritation only. There is no evidence to suggest absorption into the body systems from this contact; however, irritation will result. The degree of irritation and the time to complete recovery varies with the particular imine. The irritations induced by MAPO in tests with rabbits subsided in less than a week.

Inhalation is a minor problem. There is no data available to establish any extreme hazard, but caution should be used. The material is relatively non-volatile, although vapors may be released upon heating or during cure reaction (Refs. 2, 14, 15, 16). In well ventilated areas, no respirator system is required. When imines are used in confined areas, an air line or organic cartridge respirator should be utilized. The lower molecular weight imines (such as HX-760) have maximum allowable concentration of 200 PPM (Ref. 17).

#### CHEMICAL REACTIONS OF IMINES

In the production of solid propellant, three particular reactions of the imine are of concern. These reactions are: The reaction between the imine and the carboxyl or acidic group of the binder polymer, the reaction of the imine with water, and the reaction of one molecule of the imine with another to form a homopolymer. Only the first reaction is desirable; the other reactions are undesirable in that they can lead to a hazardous situation.

The mechanism for cure of the liquid carboxyl terminated polymer, through reaction with an imine such as MAPO, proceeds as shown at the top of the following page. The reaction between carboxyl groups of the polymer and the imine continues until a high molecular weight solid polymer results. This reaction can be carried out safely at



elevated and at ambient temperatures, but the maximum temperature should be restricted to avoid homopolymerization of the imine. The cure can be catalyzed by use of strong acid mediums although this is not advisable. The reaction changing the polymer from a liquid to solid state occurs with no significant exotherm.

The reaction of an imine with water is of importance for several reasons: (a) The reacting of the imine with excessive amounts of water will utilize available reactive groups and produce a propellant with less than desirable mechanical properties; (b) the imine may react to a degree with moisture on the skin or in the lungs inducing some of the effects noted under Physiological Properties; (c) the final product is moisture sensitive and excessive exposure may result in loss of propellant mechanical properties with ultimate grain failure. This reaction is greatly accelerated by high temperatures or acidic conditions.

The most important reaction to be guarded against is homopolymerization. While MAPO can be distilled under reduced pressure, a highly exothermic reaction can occur at atmospheric conditions. This exothermic reaction is probably in part responsible for fires that have occurred to date with this material. When the fires have involved an oxidizer, such as ammonium perchlorate, in addition to MAPO, the probability is that homopolymerization has been only a part of the reaction. When the imine is diluted sufficiently with polymer, this does not appear to be a problem.

## INCIDENTS INVOLVING MAPO

The use of MAPO in solid propellant manufacture warrants strict adherence to established safety procedures. Basically these procedures call for the dilution of MAPO with a compatible material before addition to a mixture containing ammonium perchlorate. This requirement is based upon conclusive empirical evidence that a mixture of MAPO and ammonium perchlorate can be expected to deflagrate violently at temperatures commonly encountered in the mixing of composite propellants. A series of incidents ranging from small laboratory fires to fires during the large scale mixing of propellant have occurred. In June of 1961, the Reaction Motors Division of Thiokol Chemical Corporation experienced a minor explosion (Ref. 18) attributed to the addition of MAPO to a perchlorate oxidizer. In March of 1963, a severe fire at Elkton Division of Thiokol Chemical Corporation (Ref. 22) resulted in the loss of one life (See Figures 3-10, incl.). In September of 1963, a fire occurred in a 25-gallon vertical mixer at the Rocketdyne Plant in McGregor, Texas (Ref. 19). In the incident at Elkton, the fire occurred 2-4 minutes after the addition of MAPO to the propellant mix. Investigations into the fire at the Elkton Division lead to the conclusion that the fire was caused by reaction between the curing agent and unwetted oxidizer. Laboratory experiments (Ref. 21) were made to reconfirm these findings and to establish guidelines for future operations involving MAPO and imines in general. These tests, the findings, and the results of additional compatibility studies conducted at the Huntsville Division are reviewed briefly. The Huntsville Division of Thiokol Chemical Corporation reported the following:<sup>1</sup>

"The initial evaluation of MAPO as a curing agent for carboxyl modified butadiene polymers included thermal stability tests of (1) MAPO and ammonium perchlorate, and (2) composite propellants containing MAPO. Propellants containing ammonium perchlorate, aluminum, HA polymer or HC polymer and MAPO were found to be thermally stable when cured and conditioned at 230°F. Mixtures of MAPO and ammonium perchlorate exhibited no visible reaction during 48 hours at ambient conditions but did ignite within 30 minutes at 230°F.

"Four 50 gram samples of a single lot of MAPO containing 10, 25, 50 and 75 percent ammonium perchlorate were placed in a 230°F steam table. These samples ignited and burned after approximately 20 minutes. Further compatibility studies

<sup>1</sup>Memorandum from Mr. Edgar M. Simmons, Redstone Division, TCC, to Dr. T. A. Neely, January 11, 1961

were made to determine the temperature necessary to initiate this decomposition. Samples were placed in the steam table and heat applied. Sample temperature was recorded from thermocouples imbedded in the center of 50-gram samples. The last recorded temperatures before ignition and the times required to reach those temperatures after application of heat are recorded below:

<u>Percent</u> <u>Ammonium Perchlorate</u>	<u>Temperature (°F)</u>	<u>Time (minutes)</u>
75	150	10.0
50	190	13.0
25	200	16.0
10	218	22.5

"The temperatures quoted here are not necessarily the ignition temperatures; however, a rapid increase in temperature occurred at this point which caused ignition. Two mixes containing 90 and 99 percent ammonium perchlorate failed to decompose after three hours at 230°F."

As a result of these initial studies, P. O. Blackwell (Ref. 21) reported on additional laboratory studies in which he studied the ternary system of MAPO-Ammonium Perchlorate and carboxyl terminated polybutadiene polymer at temperatures up to 230°F to develop safe techniques for adding MAPO to HC propellant mixes. Numerous fifty-gram samples containing various percentages of the ingredients were tested in steam tables. The results of this study are summarized in Figure 11. It indicates that concentrations above twenty percent of MAPO in contact with ammonium perchlorate result in spontaneous combustion when heated. The dilution of MAPO with polymer reduced the tendency to autoignite, and showed that a safe zone occurred whenever the polymer was greater than 60 percent of the liquid mixture. The following tables show sample composition, ignition temperature, and time to ignition for compositions studied.

TABLE I

Ignition Tests of Mixtures of $\text{NH}_4\text{ClO}_4$ and MAPO		
Sample Type	Observation	
<u>Weight Ratio of <math>\text{NH}_4\text{ClO}_4</math> to MAPO</u>	<u>Ignition</u>	<u>Time/Temperature °F</u>
90-10	No	32.0 hrs/235°F
75-25	Yes	10.0 min/150°F
50-50	Yes	16.0 min/170°F
25-75	Yes	16.0 min/200°F
10-90	Yes	22.5 min/206°F
1-99	Yes(50%)	18.0 hrs/230°F

TABLE II

Ignition Tests of Ternary System,  $\text{NH}_4\text{ClO}_4$  MAPO, ZL-434

Composition		Observation	
Ratio of AP to Liquids		Ignition	Time/Temperature °F
	MAPO/ZL-434		
Ratio	Blend Ratio		
50-50	1-2	No	90 hrs/230°F
50-50	1-1	Yes	14 min/170°F
50-50	1-0.5	Yes	11 min/130°F

The additional compositions, shown graphically in Figure 11 but not tabulated above, did not ignite after 18 hours at +230°F. On the basis of these studies, it was recommended that MAPO be blended with polymer in a weight ratio of at least 1-3 and that higher ratios be used when processing conditions permit.

Following the incident at Elkton, a number of lab tests were performed at 200°F, 180°F, and 160°F. Fifteen grams of fine ground ammonium perchlorate was placed in a test tube and preheated on the steam table for at least thirty minutes at predetermined temperature. Various quantities of MAPO, 0.5 to 1.5 grams, were then poured into the test tube allowing the material to flow down the side of the tube and come into contact with the ammonium perchlorate without attempting to mix the materials. The first test performed at 200°F used one gram of MAPO. Ignition occurred after one minute and forty-three seconds with no visual warning signs. The resultant flame was approximately 18 inches long and five inches in diameter (Figure 12 shows a flame of comparable dimensions.). The test was repeated and ignition occurred in one minute and forty-eight seconds. In each case approximately one-third of the ammonium perchlorate was spent. Further studies were performed with only four grams of ammonium perchlorate. The results of these studies are shown on Table III and Figures 13 and 14.

Ferric oxide was added to the ammonium perchlorate and ignition times were longer, possibly due to a dilution of the solids.

As a result of these tests, the following recommendations have been made and are followed as closely as possible:

1. Where possible, the MAPO should be incorporated (diluted) with polymer in the premix phase.
2. Where premix is not possible, remote addition of the material to the mix should be established.



TABLE III

Time to Ignition (seconds)

Concentration, Percent MAPO/Ammonium Perchlorate	Test Temperature °F				
	200	180	170	160	140
5/95	72	370	1800+	1800+	7200+
10/90	47	395	355	1800+	7200+
15/85	59	363	270	1800+	7200+
20/80	52	306	280	1180	7200+
25/75	53	378	330	-	7200+
30/70	106	312	318	-	7200+
50/50	134	427	321	-	7200+
70/30	96	462	324	-	7200+
75/25	164	527	400	-	7200+
80/20	220	440	537	-	7200+
85/15	223	528	754	-	7200+
90/10	285	2400+	552	-	7200+

NOTE: At 200°F, ignition occurred between forty-seven seconds and four and three-quarters minutes. At 180°F, the time increased from five to forty minutes. At 160°F only one sample ignited; this required 19 minutes.

3. Undiluted MAPO should not be allowed to come in contact with the mixer walls.
4. Undiluted MAPO should not be allowed to come in contact with highly acidic materials.

While these procedures are followed, no problems are anticipated as evidenced by the millions of pounds of propellant produced safely by Thiokol using MAPO and other imine cure agents. It should be made clear that while the reactivity of MAPO has been made the center of attention, other imines may be more stable or less stable than MAPO, and each should be judged on its own merit after thorough laboratory testing.

#### IN-PLANT HANDLING PROCEDURES

The use of imine curing agents at the Longhorn Division of Thiokol Chemical Corporation has been limited. Our experience as of this date has not involved propellant manufacture using the imine materials. As a result we have encountered only those situations related to proper mixing, application, and cure of liners and insulations.

These operations pose primarily those hazards classified as physiological. Up to this time, no problems have been encountered with personnel performing the necessary handling operations. This reflects that caution has been exerted in recognition of the potential problems.

MAPO, for example, is received in five-gallon containers and, once in plant, becomes the responsibility of the Chemical Laboratory. The material is analyzed for purity against raw material specifications and transferred into one-gallon polyethylene containers for convenient handling and subsequent storage. This minimizes any loss through spillage. The transfer to polyethylene containers serves the further purpose of eliminating contamination such as rust that might form on a drum.

When MAPO or other imines are required in Production, individual kits are prepared with the required amount of cure agent for the particular liner or insulation batch. Clean polyethylene bottles are used for each dispensing. These kits are prepared once a week by experienced laboratory personnel and sent to Production, based upon their scheduled requirements. These kits are then maintained at reduced temperatures until ready for use.

The mixing bay where MAPO is formulated into the end product is serviced by a controlled humidity (less than 50% R.H.) air moving system. This bay is marked "MAPO - Use Area" with limited personnel access. Prior to use, the container of MAPO is verified as correct by the Production operator and a Quality Control Inspector. After use of the MAPO, the bottles are returned to the Laboratory where they are flushed with water, drained, washed, and dried for reuse. This procedure holds personnel exposure to a minimum, and at the same time exerts a maximum control over MAPO storage and handling.

During the application of the liner and insulation materials containing MAPO, or during subsequent operations that may involve spraying of the imine as a wash, similar restrictions are imposed. These precautions are best summarized in the following instructions abstracted from Safety Bulletins issued by the Longhorn Division Safety and Security Department. These precautions are incorporated into pertinent S.O.P.'s.

## I. GENERAL SAFETY PRECAUTIONS

- A. Personnel involved in operations with MAPO shall wear coveralls, neoprene gloves and splash-proof goggles.

NOTE: 1. Gloves shall be free of holes and must be thoroughly washed with soap and water after use.

2. All clothing and accessory safety equipment contaminated with MAPO and/or materials containing MAPO shall be stored in a separate container marked "MAPO Contamination."

- B. Work in well ventilated working areas.
- C. Do not carry articles which might come in contact with the mouth while handling MAPO and/or materials containing MAPO.
- D. After handling MAPO and/or materials containing MAPO, personnel should wash the face and hands thoroughly with soap and water prior to eating.
- E. Clothing contaminated with MAPO and/or materials containing MAPO should be changed as soon as practical.
- F. Containers of MAPO and materials containing MAPO shall be clearly labeled "MAPO" or "Materials contain MAPO".
- G. Immediately after MAPO or materials containing MAPO have been removed from a container, the container shall be flushed with large volumes of water and thoroughly washed with soap and water.
- H. Bags and equipment contaminated with MAPO or material containing MAPO shall be plainly marked until cleaned.
- I. Areas and equipment contaminated with MAPO or material containing MAPO shall be thoroughly washed with soap and water.
- J. When disposing of MAPO, dilute with large volumes of water.
- K. Personnel who have handled MAPO or materials containing MAPO and have an unexplained illness shall report for First Aid immediately.

## II. SPECIAL SAFETY HAZARDS AND PRECAUTIONS

- A. Avoid combining raw MAPO with oxidizer, e.g.,  $\text{NH}_4\text{ClO}_4$ ,  $\text{N}_2\text{O}_4$  or other compounds acidic in nature, e.g.,  $\text{H}_2\text{SO}_4$ , etc. Since this constitutes a definite fire and explosive hazard.
- B. After raw MAPO has been diluted within a 4:1 (Liquid:MAPO) ratio, it no longer presents a fire or explosive hazard.
- C. Personnel involved in the spray application of MAPO shall be kept to a minimum, e.g., for spraying interior of a case.

NOTE: Limit operators to 3 and transients to 2.

- D. Anyone within five feet of the motor case during spray application must comply with the following:
  - 1. Wear a fresh air respirator.
  - 2. Wear full face shield.
  - 3. Take a shower immediately after completion of spray application.
  - 4. All clothing and protective safety equipment shall be washed before re-use.

## III. ANTIDOTAL PROCEDURE

- A. If accidentally swallowed, induce vomiting and obtain medical attention immediately.
- B. In case of contact with the skin, immediately flush the affected area with water and wash thoroughly with soap and water.
- C. In case of contact with the eyes, flush thoroughly with water and obtain medical attention promptly.

## TECHNIQUES FOR IMINES

As with any new material, the use of imines in Production operations is preceded by stringent laboratory testing. The methods developed during these investigations are primarily for control of raw materials and end product. Many of the techniques developed can be put to good use in monitoring levels of imines or detecting contamination when necessary.

The pure imine as received can be characterized using the IR Spectrophotometer. Any subsequent contamination can be determined by a re-analysis and comparison of the spectra. Any change in the characteristic bands shown is indicative of contamination or a reaction having occurred. This same technique is useful when area contamination with an imine is suspected. A wipe test of the area in question is made using a solvent-treated pure gauze; the resultant solution is tested on the spectro-photometer against a previously calibrated standard. Any residual imine on equipment or other facilities is easily determined.

When it is desirable to determine the concentration in a vapor phase either the gas chromatograph or IR Spectrophotometer can be used. A sample of the vapor is collected in a calibrated gas cell, and analyzed with either instrument. This permits a quantitative analysis of decomposition vapors, concentration of vapors in an operating area, and determination of use/storage temperature limits.

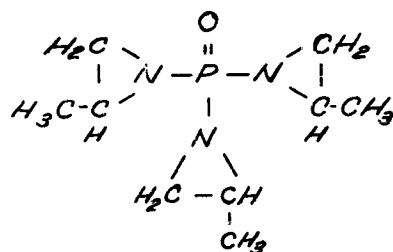
When it is desirable to check the compatibility of an imine with other materials, the differential thermal analysis unit is used. With this method, it is possible to determine not only the autoignition point, but to detect any endothermic or exothermic reactions that might be undesirable.

Other instruments useful in monitoring imine levels would include the X-ray diffraction unit. The use of any of the above instruments should provide an adequate control and monitoring system.

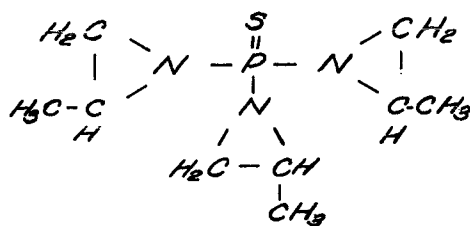
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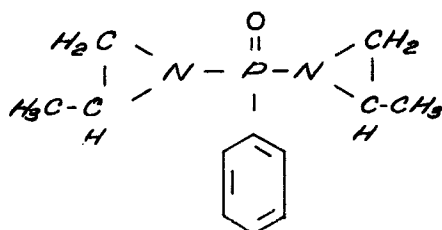
FIGURE 1  
MAPO and MAPO-Type Cure Agents



MAPO

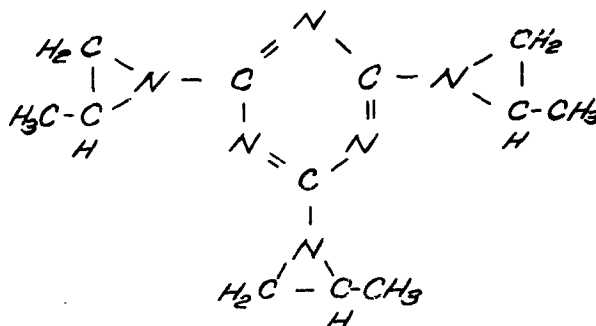


MAPS

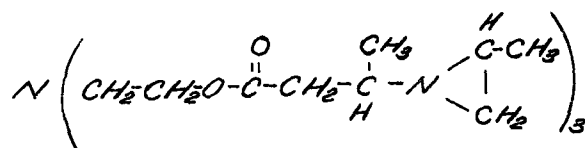


Phenyl MAPO

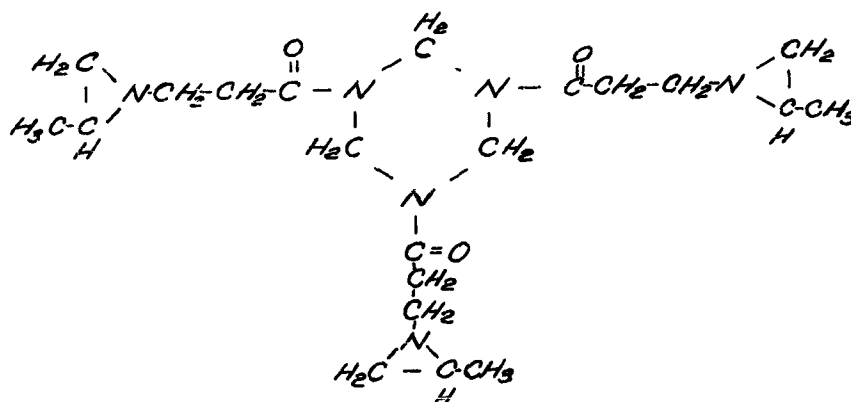
FIGURE 2  
Typical Imine Cure Agents



Tripropylenemelamine



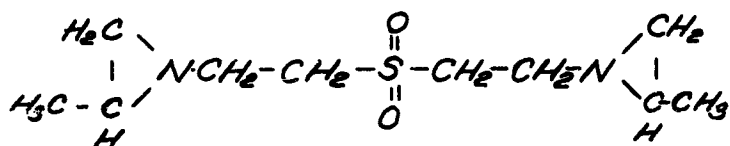
Nitritotriethyl-Propyleneiminobutyrate(NTPB)



Tris 1,3,5(2-Methylaziridinylpropionyl)Hexahydro-1,3,5-Triazine



Figure 2 cont'd.



Bis (2-Methyl Aziridinyl Ethyl) Sulfone  
ZC-466

FIGURE 3  
BUILDING DAMAGE, ELKTON FIRE



FIGURE 4

MIXER CUBICLE, ELKTON FIRE

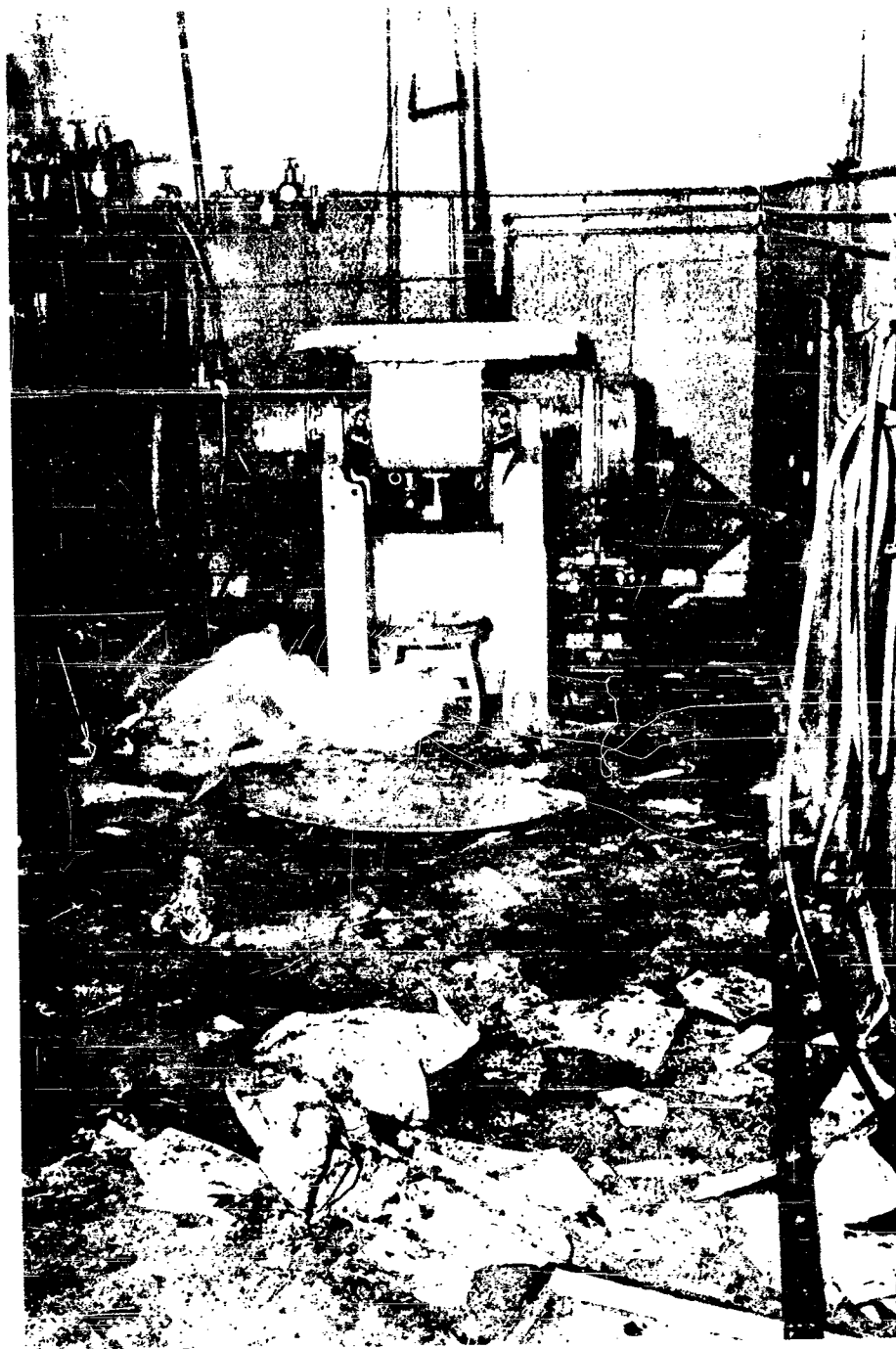


FIGURE 5  
INTERIOR OF MIXER, ELKTON FIRE

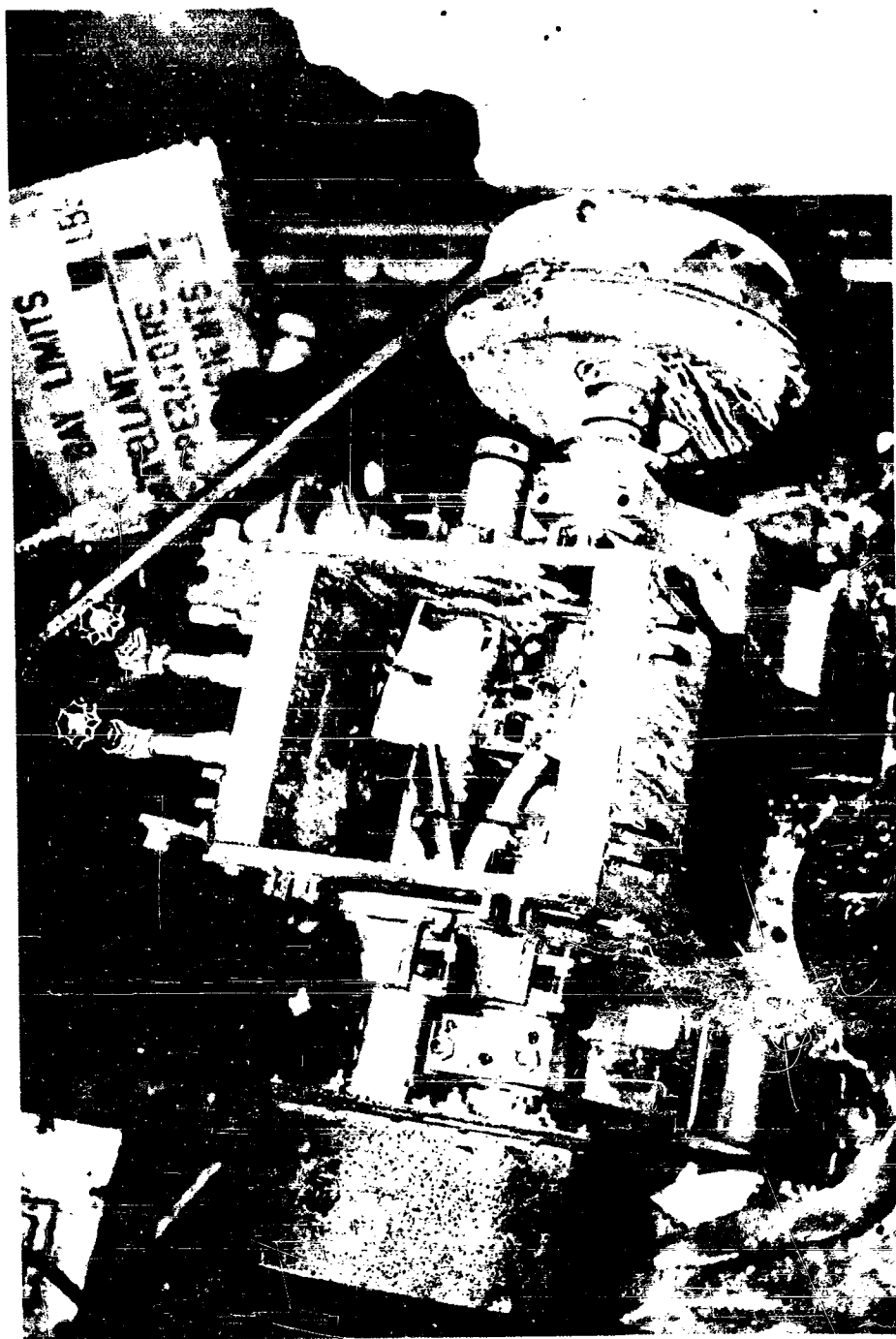


FIGURE 6

WEST WALL OF MIXER CUBICLE, ELKTON FIRE



FIGURE 7

EAST WALL OF MIXER CUBICLE, ELKTON FIRE

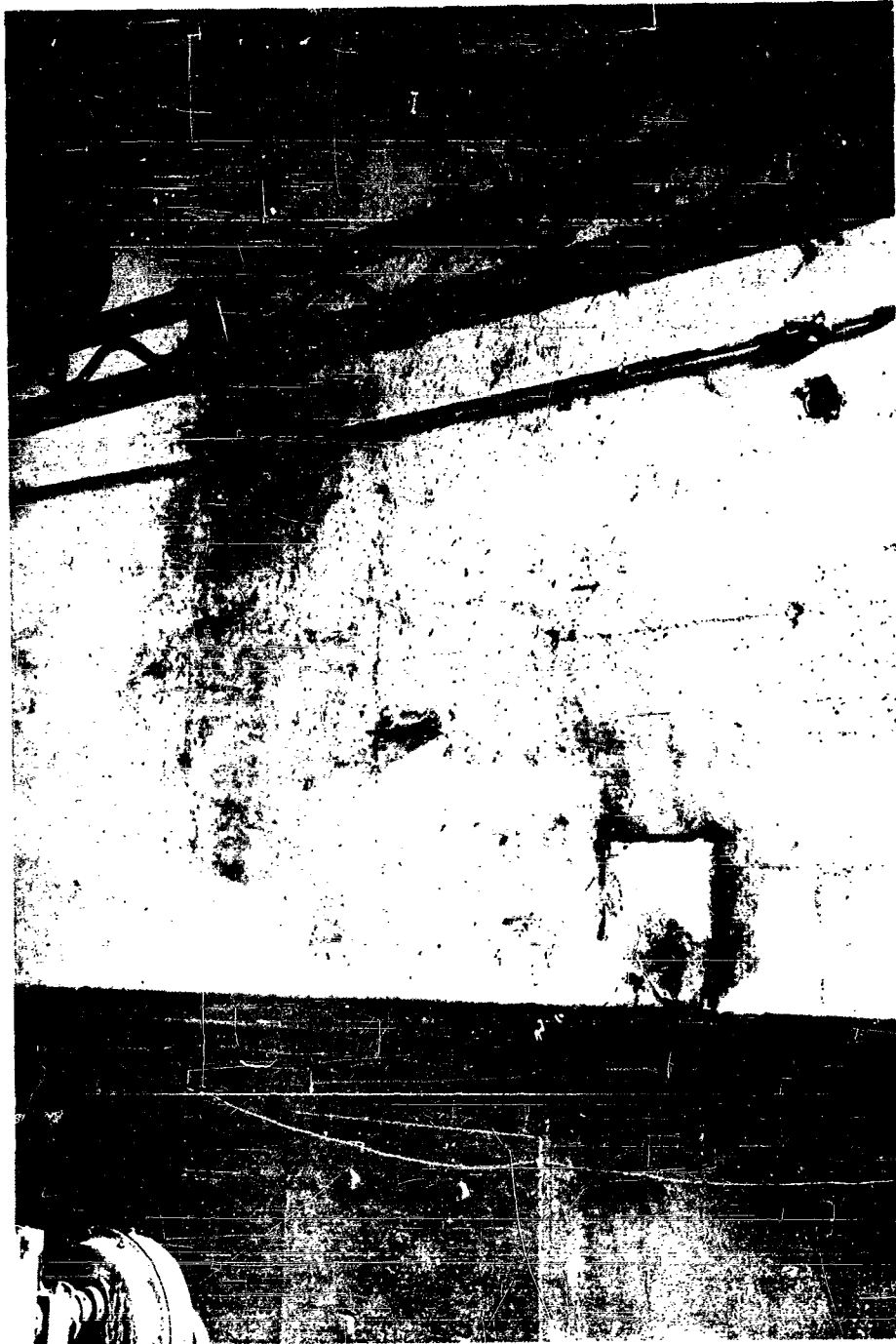


FIGURE 8  
BOTTOM OF MIXER BOWL, ELKTON FIRE



FIGURE 9  
TOP OF MIXER BLADES, ELKTON FIRE

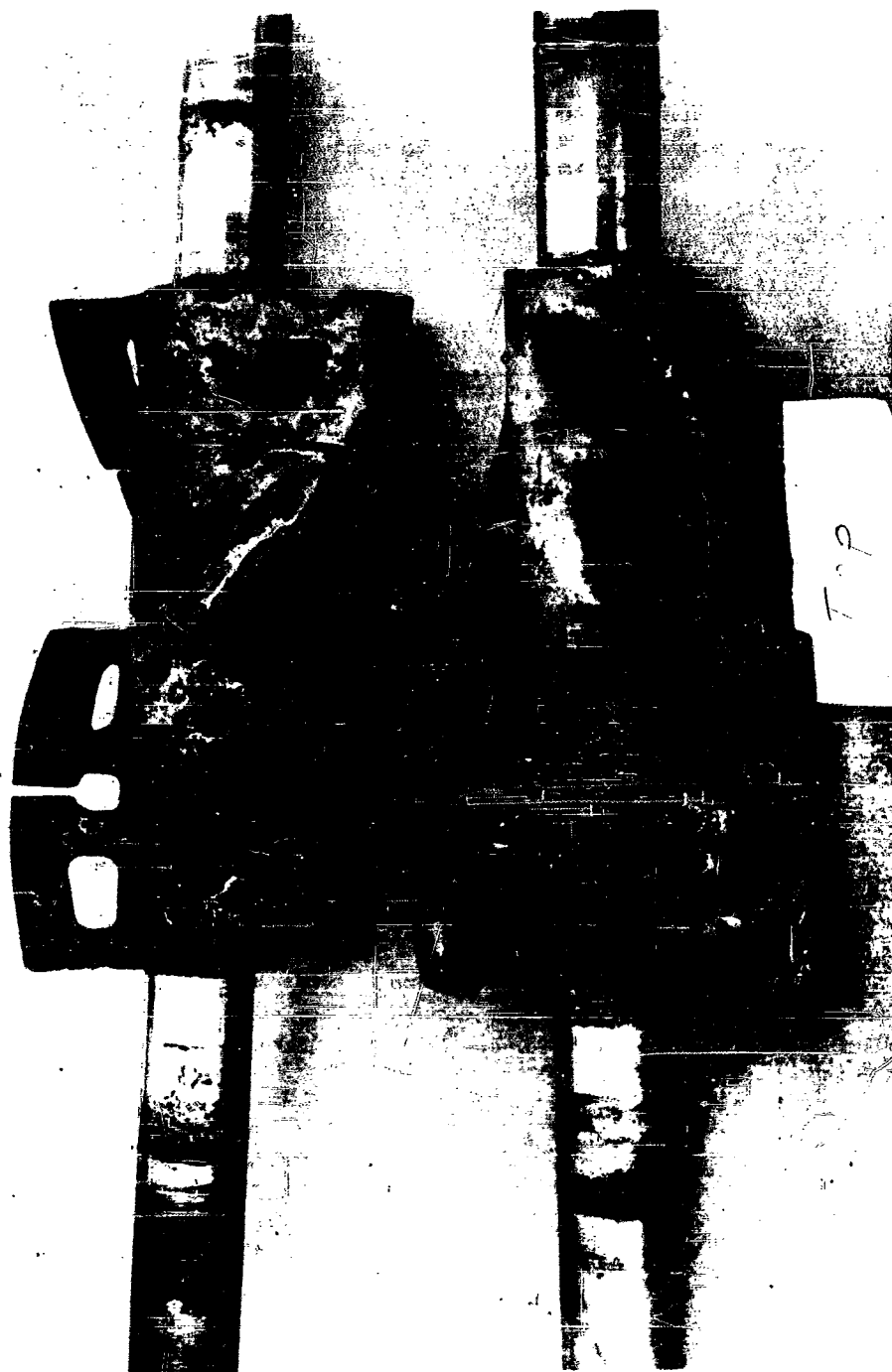




FIGURE 10  
SPATULA, ELKTON FIRE

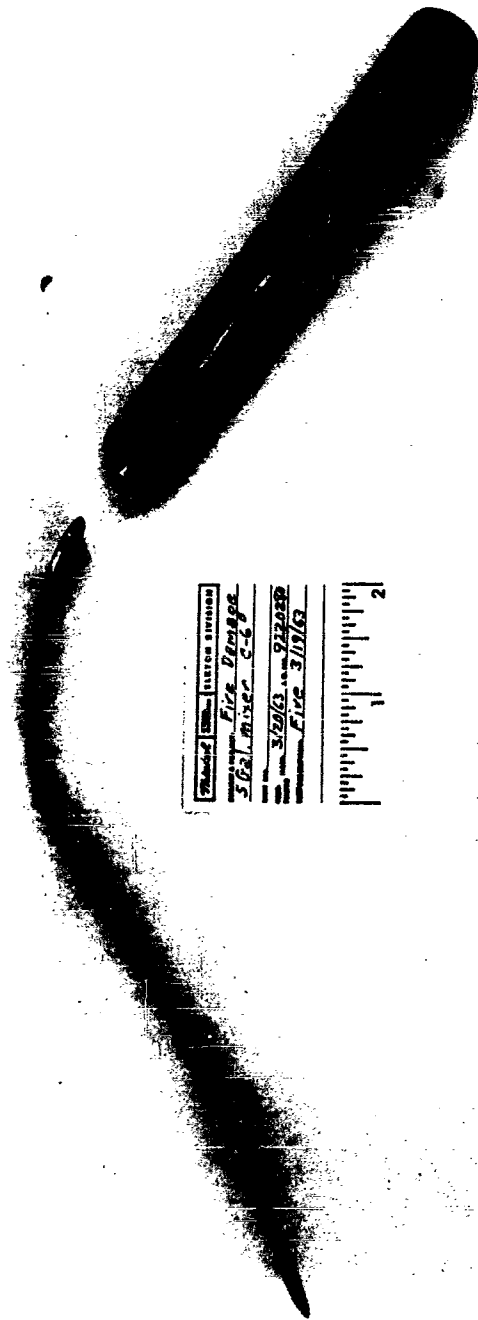


FIGURE 11

TERNARY SYSTEM STUDY

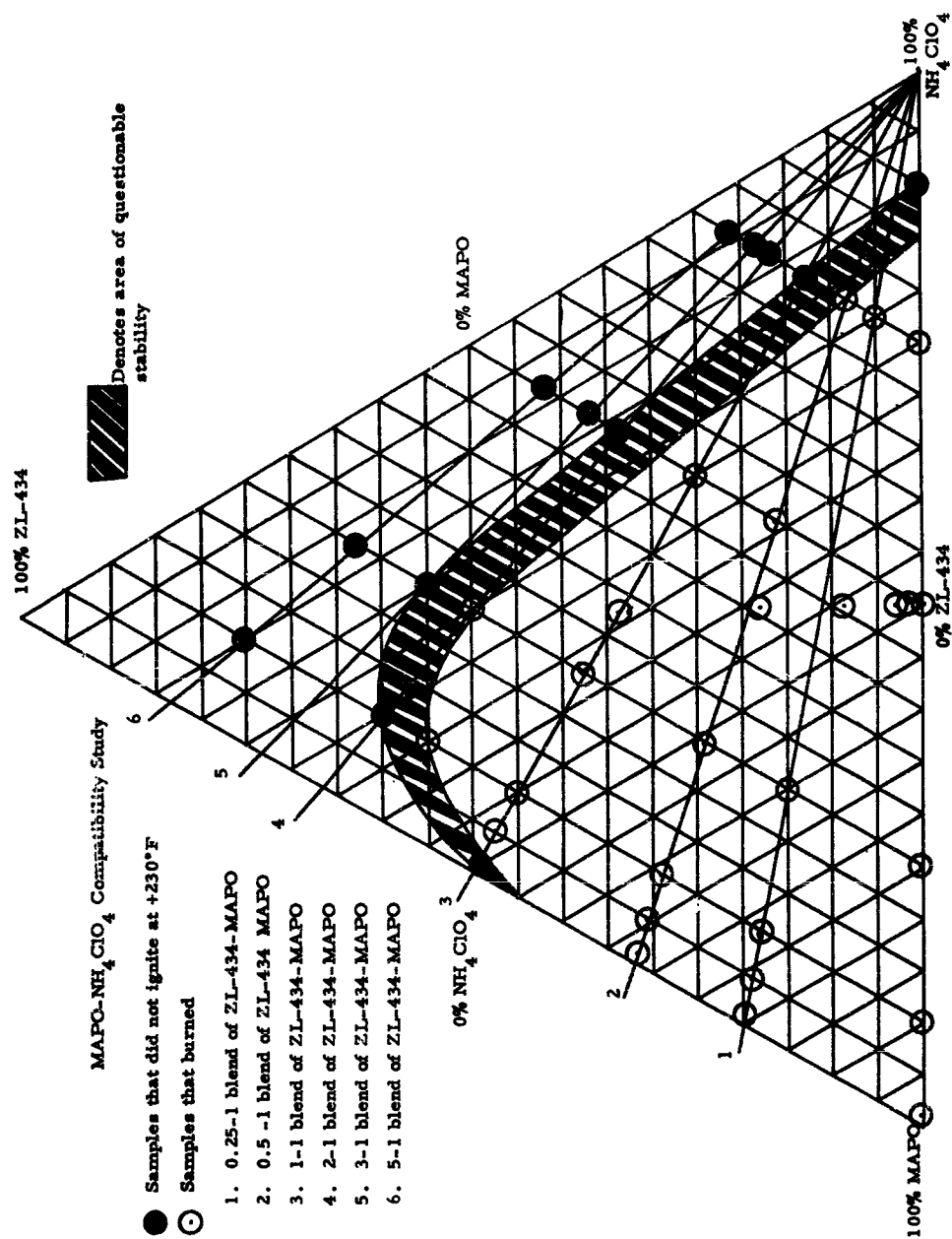


FIGURE 12  
REACTION BETWEEN MAPO AND UNCOATED AP

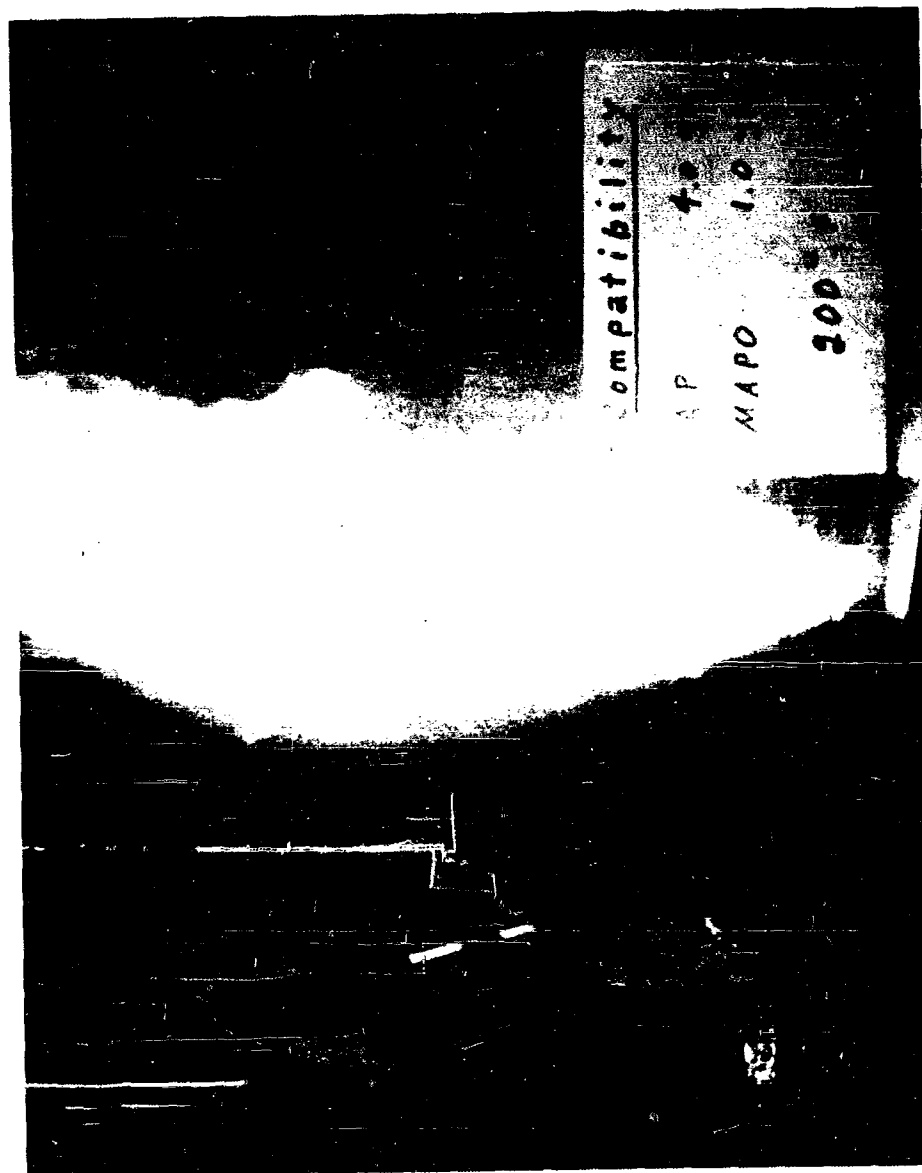
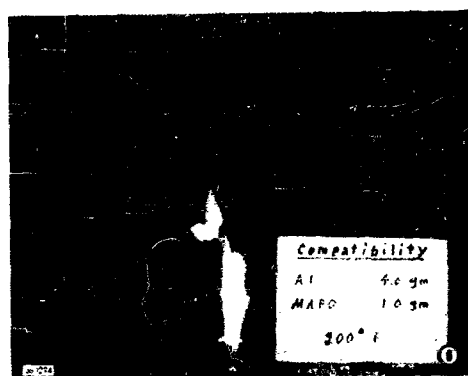
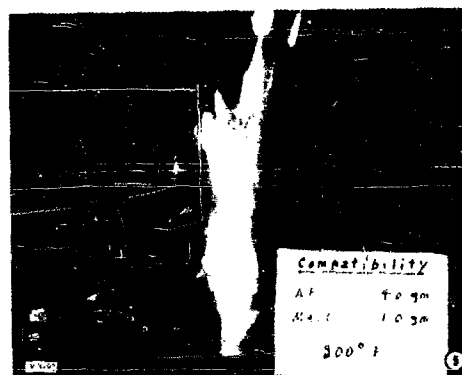
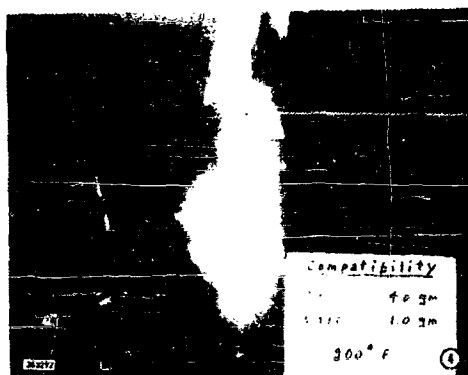
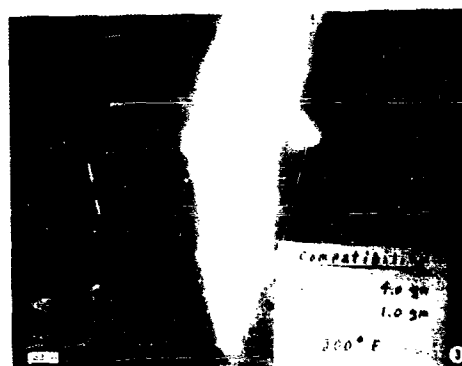
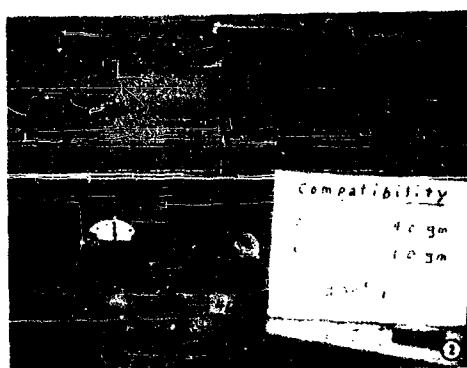


FIGURE (13 and) 14

MAPO/AP COMPATIBILITY TESTING



**FIGURE 13 (and 14)**  
**MAPO/AP COMPATIBILITY TESTING**



## DEVELOPMENT OF SAFETY CONTAINERS AND THEIR EVALUATION

Dr. M. Zimmer, U. S. Naval Propellant Plant

Inspired by Du Pont, this study was initiated to develop an explosive safety container able to withstand the explosion of higher loads of nitroglycerin than 2 g and to investigate the behavior of the container during an explosion.

To be detonation-safe, the sample container was required to withstand at least a minimum load of explosive material. To be additionally safe, the sample container (Figure 1) was to be fabricated from a material which would form harmless fragments upon break-up from a maximum load detonation.

Initially, an exploratory study was made with polyurethane containers. Polyurethane material was chosen because: (1) it was available, (2) it met most of the prerequisites for a good safety container, and (3) it was a transparent material so that the effects of explosion could be observed throughout the structure. From the results of this study, other investigations concerning the improvement of the polyurethane containers were carried out. Various other materials were analyzed for possible use for containers.

### RESULTS

Polyurethane explosive sample containers were fabricated with 40% toluene diisocyanate (80/20 of 2,4-/2,6-) and 60% castor oil.

As an initial step, the sample containers were filled with a 1- to 5-g load of nitroglycerin. It was decided to use a small cyanuric triazide squib (50-70 mg) with 12.5 joules of electric energy to set off the nitroglycerin. It was assumed that any accidental detonation would be initiated by a relatively small quantity of energy in the form of shock, electric-static energy, local heating effects, etc.

As expected, the "detonation" was of low order. The nitroglycerin reacted in a few microseconds within the short confines of the vial. This low-order reaction will henceforth be termed an "explosion" rather than a "detonation."

Figure 2 shows polyurethane safety containers after testing with 1, 2, 3, 4, and 5 g of nitroglycerin.

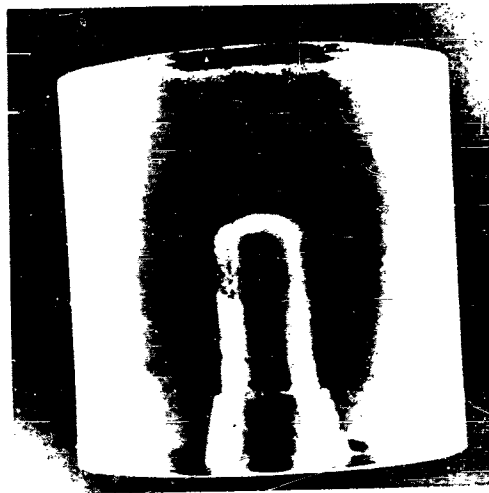
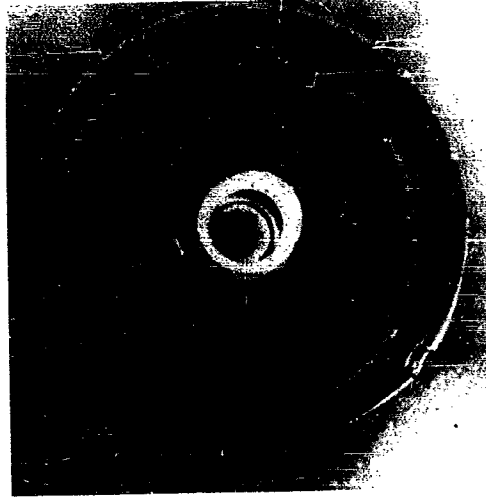


Figure 1 - Top and side view of a polyurethane container  
(dia., 8.1 cm; height, 8.1 cm)

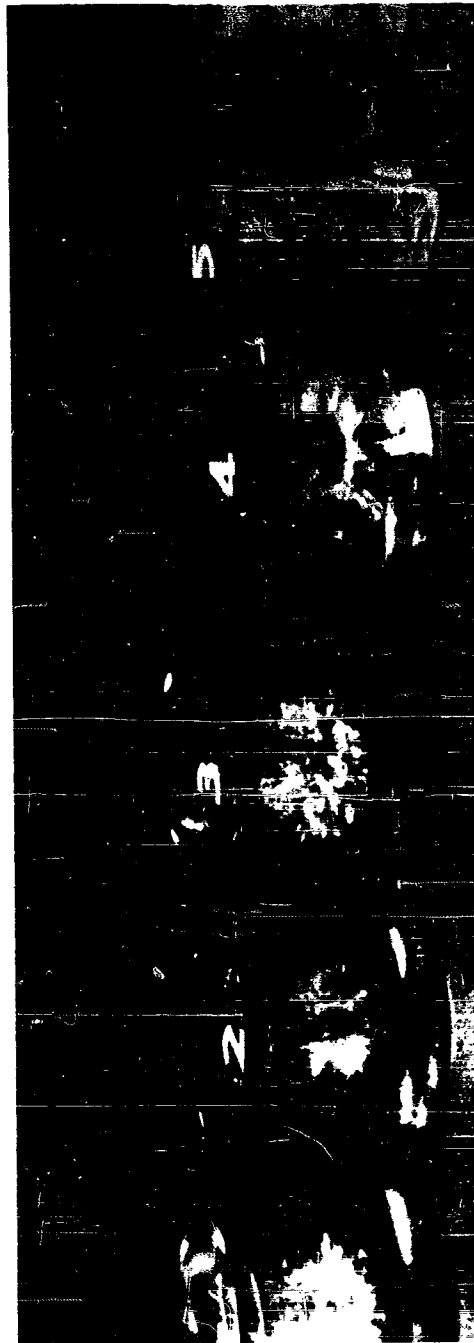
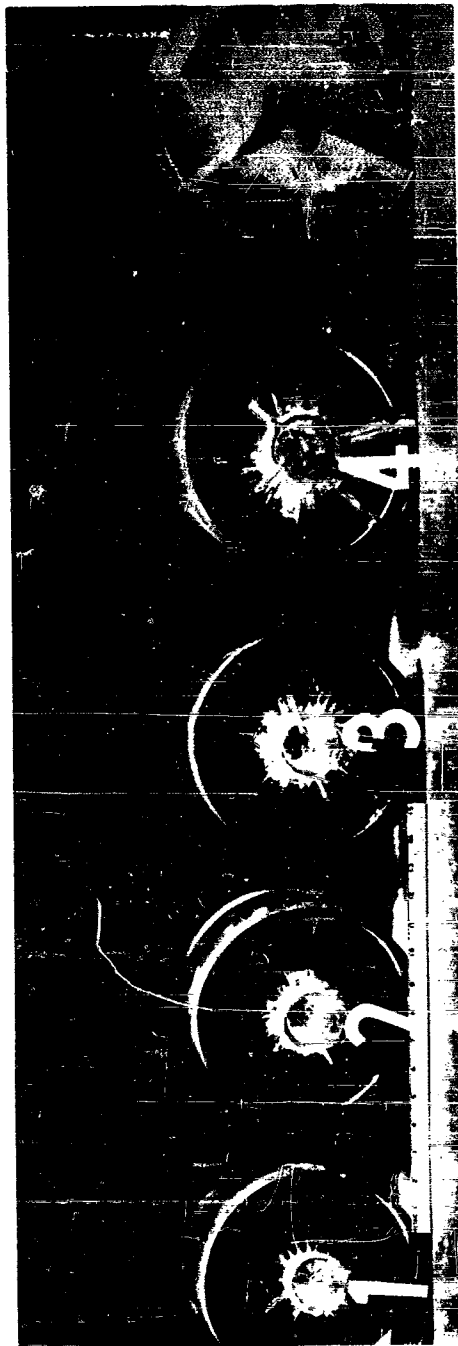


Figure 2 - Polyurethane safety container after testing with several loads of nitroglycerin; detonator, approx. 75 mg cyanuric triazide; No. 1, 1 g nitroglycerin; No. 2, 1.5 g nitroglycerin; No. 3, 2 g nitroglycerin; No. 4, 4 g nitroglycerin; and No. 5, 5 g nitroglycerin.



### Strengthening and Modifying the Polyurethane Safety Container:

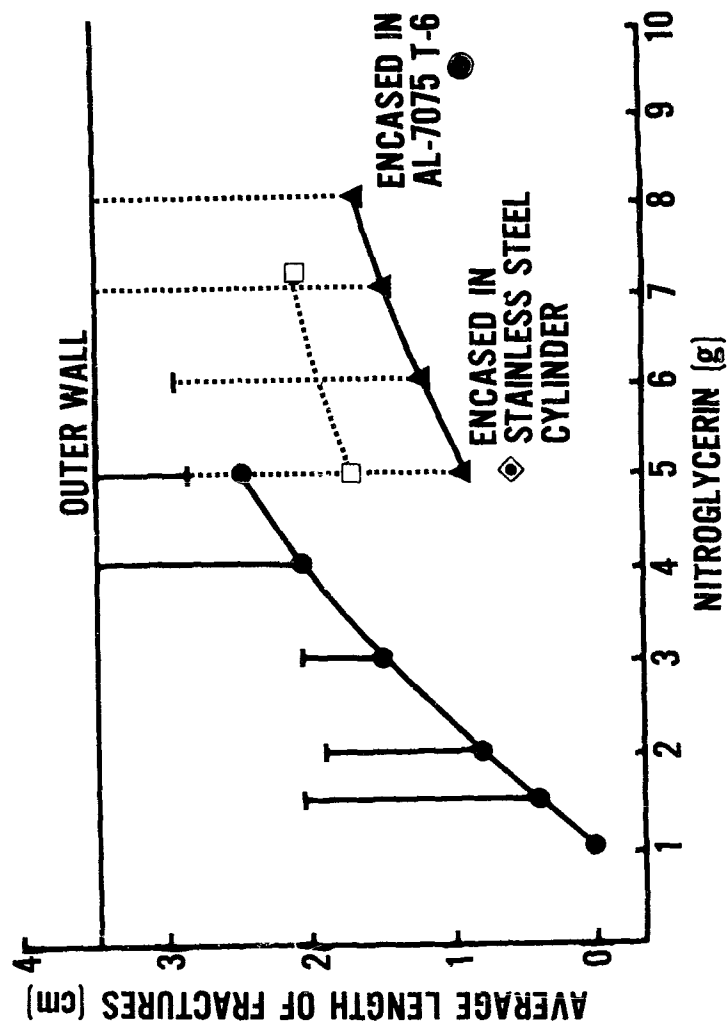
From this preliminary and exploratory study, the following question concerning the most favorable characteristics for an explosive safety container arose. Would it be possible to strengthen or modify the polyurethane container so that it could handle larger loads of explosive materials? The following modifications to the basic container were innovated and the containers were tested with 5 g of nitroglycerin.

(1) Wrapping or Encasing the Container with Strong Materials: The total strength of the container can be increased by wrapping it with a strong cord or wire, or by encasing it with a strong metal. The polyurethane, around the central vial, is prestressed by the compressive forces generated by the expanding explosion gases and, therefore, is prevented from expanding normally. Figure 3 shows the increased load-capacity for containers wrapped with 120-lb-test Dacron line and 101-lb-test nichrome wire; the Dacron-wrapped container was able to withstand a load of 8 g of nitroglycerin. Figure 4 is a picture of these containers and also one of an aluminum-encased polyurethane container after explosive testing. In order to see the damage on the containers, the strengthening material was removed after the explosion. Container 1 was strengthened with 3 layers of Dacron fishing cord, 130-lb-test and withstood the explosion of 8 g nitroglycerin, and container 2 was strengthened with an aluminum encasing of 125-mm wall thickness and withstood the explosion of 9.5 g nitroglycerin. The third container was fabricated from aluminum (AL-7075-T-6) and withstood the explosion of 10 g nitroglycerin with very little change in the outside dimensions. Stainless-steel-walled containers (1.8-mm thick) were able to withstand the explosion of 5 g loads, but the steel castings were deformed to some extent.

(2) Increasing Tensile Strength of Polyurethane Container by Addition of Strong Fibers: To increase the tensile strength of the material, various amounts of glass wool, glass fiber fabric, and nylon fabric were added in a spiral pattern around the central vial. Three sample containers, with each of the above mentioned fiber and fabric additions, were tested with 5 g of nitroglycerin. The results showed that the improvements in strength were not enough to warrant further investigation along this line.

(3) Improvement of the Material Properties of the Zone Surrounding the Central Vial: Attempts were made to improve the elongation and cold-flow properties of the zone surrounding the central vial. This was done by inserting various materials, e.g., natural rubber, Wood's metal, etc., in this zone. There

# **FRACTURES OF EXPLOSIVE POLYURETHANE SAFETY CONTAINER CAUSED BY EXPLODING NITROGLYCERIN**



- WITHOUT STRENGTHENING MATERIAL
- ▲ STRENGTHENED WITH 3 LAYERS OF 120-lb TEST DACRON LINE
- STRENGTHENED WITH 1 LAYER NICHROME WIRE 101-lb TEST

Figure 3

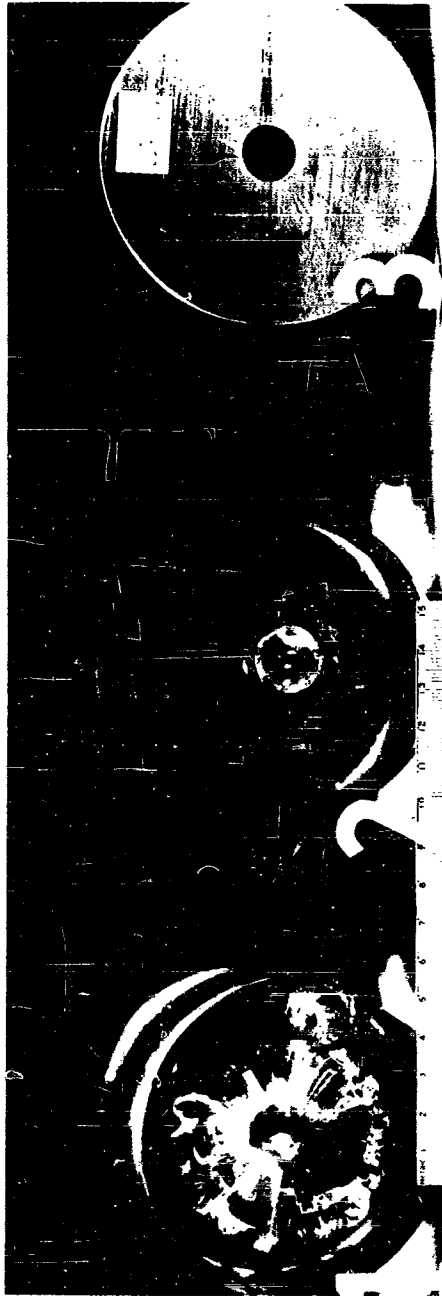
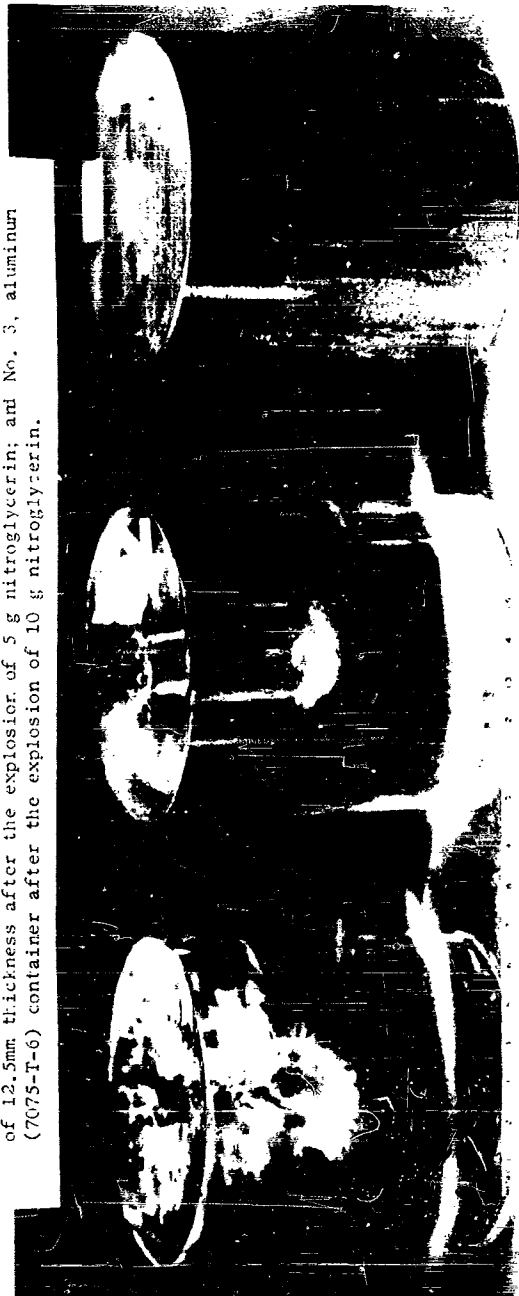


Figure 4. Polyurethane containers strengthened with No. 1, three layers of dacron cord, 130-pound test, after the explosion of 5 g nitroglycerin; No. 2, aluminum outer walls of 12.5mm thickness after the explosion of 5 g nitroglycerin; and No. 3, aluminum (7075-T-6) container after the explosion of 10 g nitroglycerin.



was some improvement in the load capacity; however, the total strength of the containers was lessened by the substitution of the polyurethane by the low-strength (but good cold-flow property) material.

(4) Interruption of the Propagation of Fracture Lines and of Shock Wave: When a material is under stress, fractures in the structure cause an over-all loss of strength. By interrupting the propagation of the fracture from the center, the container should be able to withstand the explosions of larger loads of nitroglycerin. Some containers were fabricated employing two methods listed:

An 8-cm-wide strip of 0.15-cm-thick polyurethane was wound around a 12.5-mm (inner diameter) vial until an 8-cm-diameter container was formed. A 12.5-mm plug was inserted below the vial.

Glass and polyethylene beakers and vials were positioned concentrically around the central vial, and polyurethane was molded around the tubes and beakers.

As a shock wave is partially reflected and attenuated at each interface or inhomogeneity of a material, the many interfaces created by the separated material should also attenuate the shock wave as it passes through the container. In all probability, the poor bond between the polyurethane layers of the first type container and between the polyethylene and polyurethane of the second type container weakened the structure of the containers as they did not withstand the explosion of 5 g of nitroglycerin. However, there were indications that the polyethylene interfaces prevented propagation of fractures and that there seemed to be less damage done to the polyethylene-beaker containers.

To improve the shock attenuating qualities of the polyurethane, containers with foamed polyurethane were fabricated and tested with 5 g of nitroglycerin. (Air bubbles are good shock attenuators - for example, in underwater explosions the measuring equipment is protected by surrounding it with a veil of air bubbles. A large part of the shock energy is annihilated by the air bubbles.)

In all of these containers, the structure was weakened to such an extent by the modifications to the container that they were fragmented to many pieces by the explosion of the nitroglycerin.

### Effect of Vial Material, Shape and Size on Over-All Container Strength:

Vial Material: The results from the first explosions on containers fabricated with glass vial inserts indicated that the fracture lines in the polyurethane seemed to originate from the fracture lines in the central glass vial. To control this, vials of various materials were inserted into the polyurethane containers. Besides glass, polyethylene of medium density (with its high percentage of elongation and good tensile strength) and polypropylene of high density were used as vial materials. High-density polypropylene has excellent tensile strength but a low percentage of elongation. The amount of fracture formation in the polyethylene vial container, when exploded with nitroglycerin, was considerably less than in either the polypropylene or in the glass.

Influence of the Vial Shape: Changing the shape of the vial might enable the container to withstand the explosion of higher loads of nitroglycerin by either enclosing the blast or by directing most of the blast effect upward. Therefore, polyethylene vials of the shapes shown in Figure 5 were tested in molded polyurethane containers.

For a 5 g load of nitroglycerin no observable advantage in any specific shape was found. Since the cylindrical vial was the easiest to fabricate, all containers made later had vials of this shape.

Critical Diameter of the Vial Inserted in the Safety Container: The effect of the explosion velocity on the investigated safety container can be compared to the influence of the detonation velocity on the Trauzl-Block. As high pressures in the detonation front correspond to high-detonation velocities, the detonation velocity must be taken into account.

Charge diameter and length, chemical characteristics, physical state and condition of the explosive, and the amount of detonator determine the detonation velocities of the nitroglycerin. Below 100 mg of cyanuric triazide, the amount of detonator does not appreciably change the explosion velocity for a given diameter charge.

The reaction process for the explosion with small detonators seems to be an unsteady one. The initial explosion velocity is not sustained continuously. The nitroglycerin reacts in spots down the length of the vial as the shock front passes. As generally the detonation velocity is dependent upon the inner diameter of the tube containing the charge, it was considered

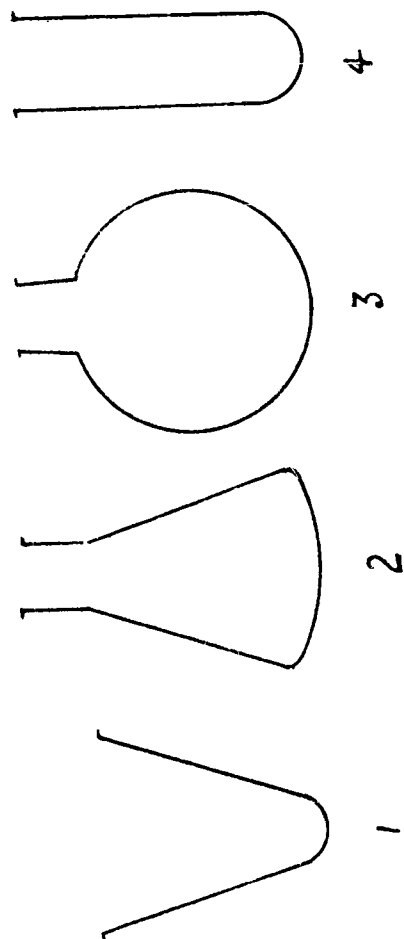


Figure 5 - Polyethylene vials of various shapes.

essential, in the case of the small (8- or 10-cm diameter) safety container, to keep the vial diameter below the "critical diameter" for nitroglycerin initiated by small detonators. This "critical diameter" was measured at 12.5 mm. At this diameter the "explosion velocity", 485 m/sec and lower for smaller diameter, rises sharply and passes into a steady state detonation of high order with increasing diameter. Figure 6 shows the influence of charge diameter on the detonation velocity of nitroglycerin.

The ratio of the diameter of the vial to the length of the charge should be kept as high as possible so that a steady plane detonation wave cannot build up as easily as it would if the charge length were high compared to its diameter.

#### Physical Structure of Safety Container:

During an explosion, there are two possible destructive forces acting on the container: (1) the shock wave, and (2) the outward pressure exerted by the hot gases from the nitroglycerin explosion. The containers begin to bulge within a few microseconds after the initiation of the nitroglycerin, and fracture lines start to form soon after - the time interval being dependent upon the nitroglycerin load. Figure 7 shows some selected framing sequences for a 5 g load of nitroglycerin.

The container elongates along the circumference as the hot detonation gases expand. The inner circumference, or wall of the container, will elongate at a much higher rate for a given radial expansion of the inner hole than a zone farther from the center periphery. Therefore, the polyurethane will start to fracture from the inner wall when maximum elongation of the material is reached.

As the explosion of the nitroglycerin proceeds, the gases will be ejected from the top of the open vial and the gas pressure will bulge the walls of the sample container. These two actions will cause the inner pressure to decrease with time. The fracture of the walls will continue until the forces acting upon the polyurethane, at that point, approach the maximum tensile strength and the maximum elongation point of the material. At this point, as the inner expanding forces are continually decreasing, the fracture will stop.

It was found that the bottom part of the container was affected much less by the destructive forces than the upper part. For instance, Figure 8 is a photograph of a safety container tested with 7 g of nitroglycerin. The walls around the central vials were peeled off and a pyramid-shaped torso

# **DETONATION VELOCITY OF NITROGLYCERIN AS A FUNCTION OF THE CHARGE DIAMETER**

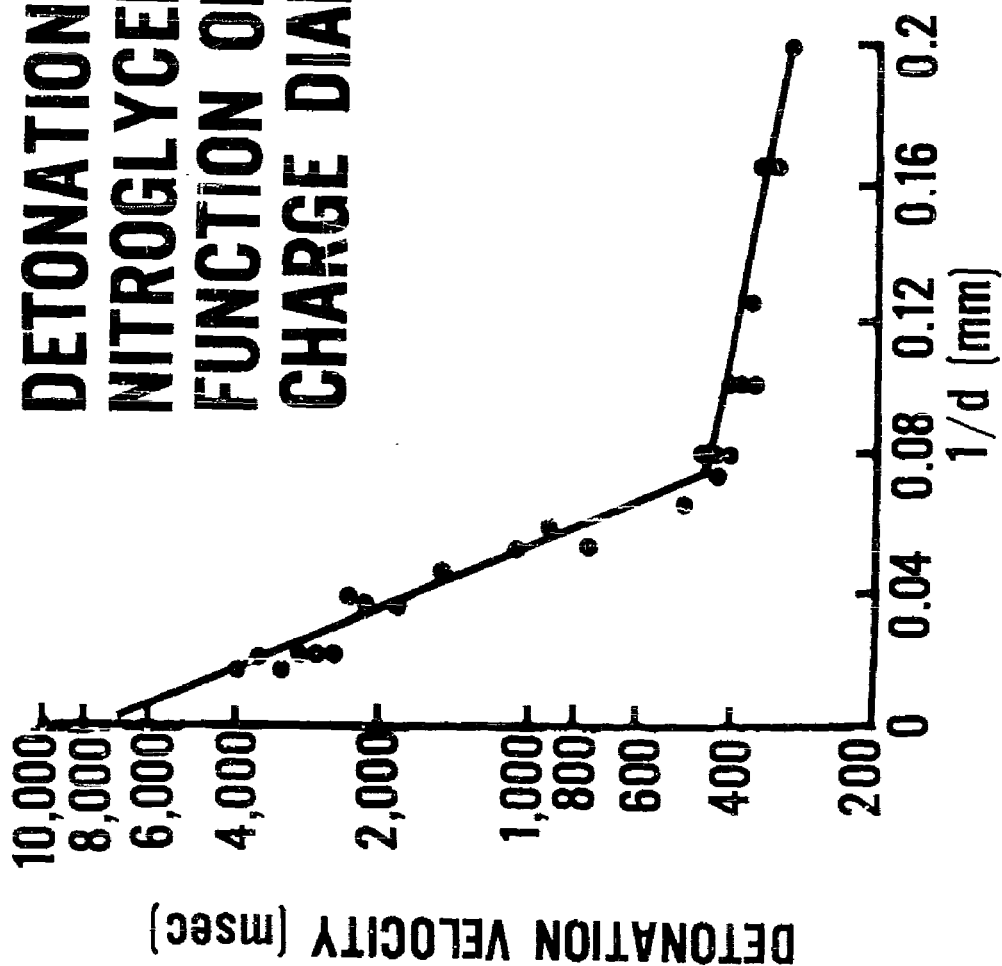


Figure 6



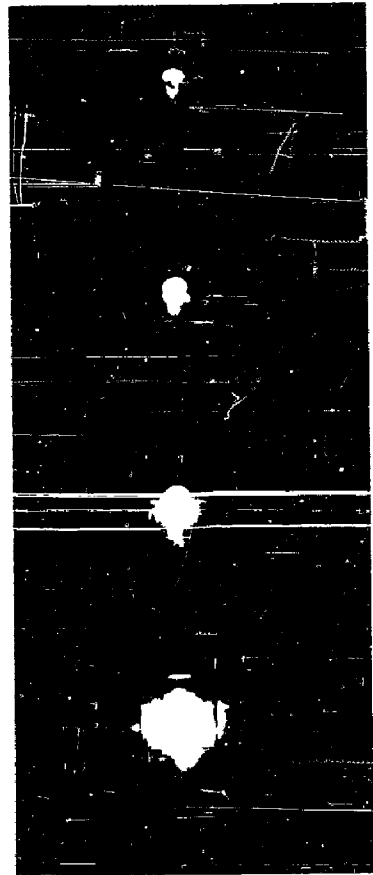
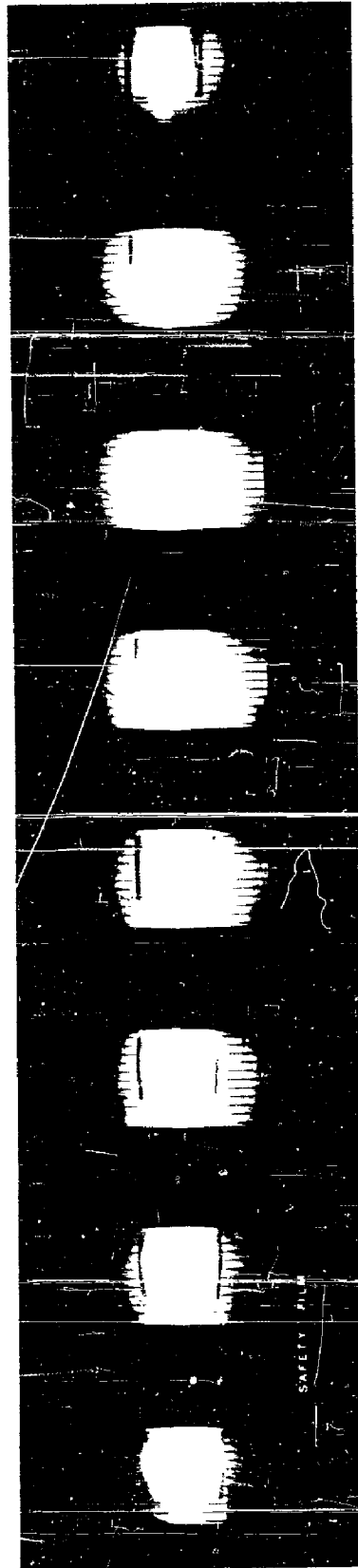
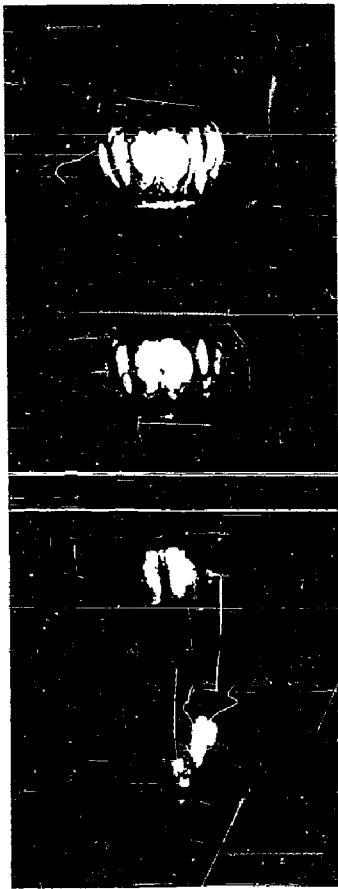


Figure 7 - Explosion of 5 g nitroglycerin in a polyurethane container with frame time of 16 usec.



Figure 8-Safety container after testing with 7 g nitroglycerin

remained, the summit of which was the bottom of the vial. Each time the tested safety container did not withstand the blast a similarly shaped torso was left. For this reason some tension-optic pictures were made to find if some stress lines had been asserted in the material by the molding procedure. A typical photograph is given in Figure 9.

In Figure 10 the safety container was set under a static pressure by forcing a wedge into the central vial. The container, being under stress, showed stress lines indicating the direction of later fracture lines when higher pressure (explosion) was applied.

In order to obtain information on polyurethane properties an investigation was made to determine shock attenuation and the equation of state of polyurethane. In these experiments two methods - one based upon the "aquarium technique" and the other upon the measurement of the free surface velocity - were used to acquire the data needed to calculate the equation of state of the polyurethane. The measured pressure region was from 1 to 200 kbars. A shock wave traveling through water can be easily and continuously observed and recorded by a streak camera. This means that water can be used as a "pressure gage" to measure transient pressures. A shock was transmitted into the water from the sample. Then the pressure in the sample was calculated from the measured shock velocity and pressure in the water. In the first method applied to this study, the shock "impedance mismatch" equation was used:

$$P = P_2 \frac{(\rho_{01}D_1 + \rho_{02}D_2)}{2\rho_{01}D_1}$$

P = pressure

D = shock velocity

$\rho_0$  = initial density

Figure 11 shows the attenuation of a shock in polyurethane as a function of distance. The ratio length to diameter has only an influence on the attenuation when it is smaller than 3.5. The attenuation of the shock pressure as a function of distance is shown in Figure 12, and Figure 13 the relation between shock pressure and shock velocity is given for polyurethane. The dotted curve indicates the same relation for lucite as reported by Cook.

Figure 14 presents a pressure-specific volume diagram for polyurethane.

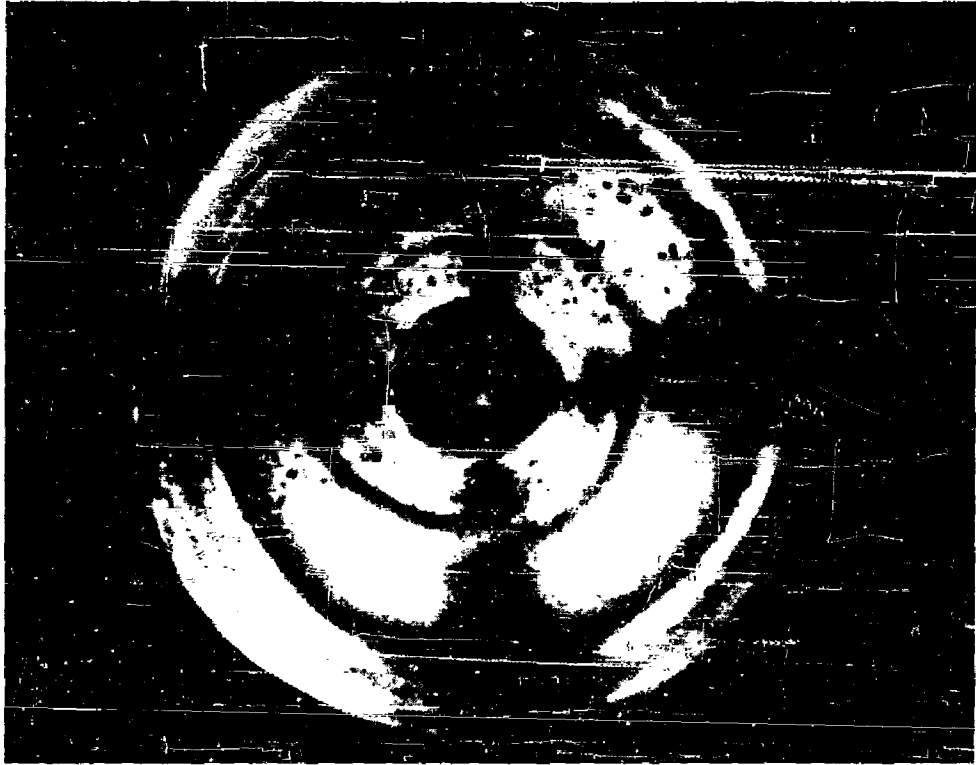


Figure 9 - Stress lines in a safety container at rest



Figure 10 - Stress lines in a safety container under pressure

# SHOCK ATTENUATION IN POLYURETHANE AS A FUNCTION OF DISTANCE AND CHARGE RATIO: LENGTH/DIAMETER

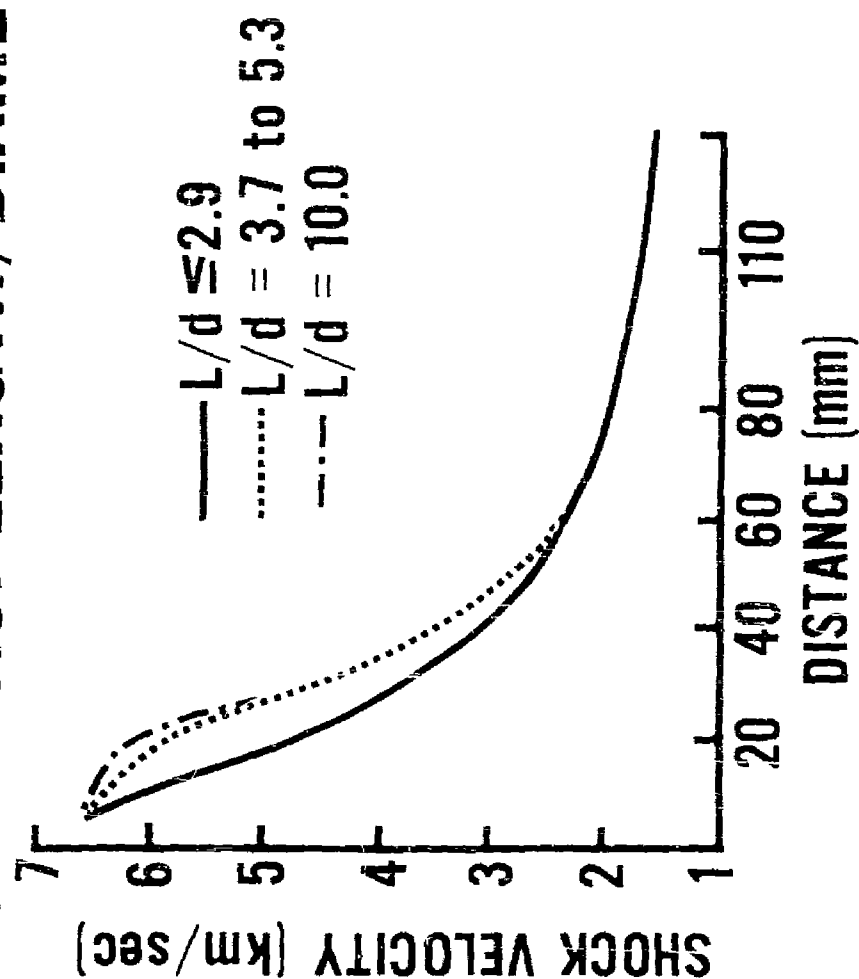


Figure 11

# SHOCK PRESSURE IN POLYURETHANE AS A FUNCTION OF POLYURETHANE THICKNESS

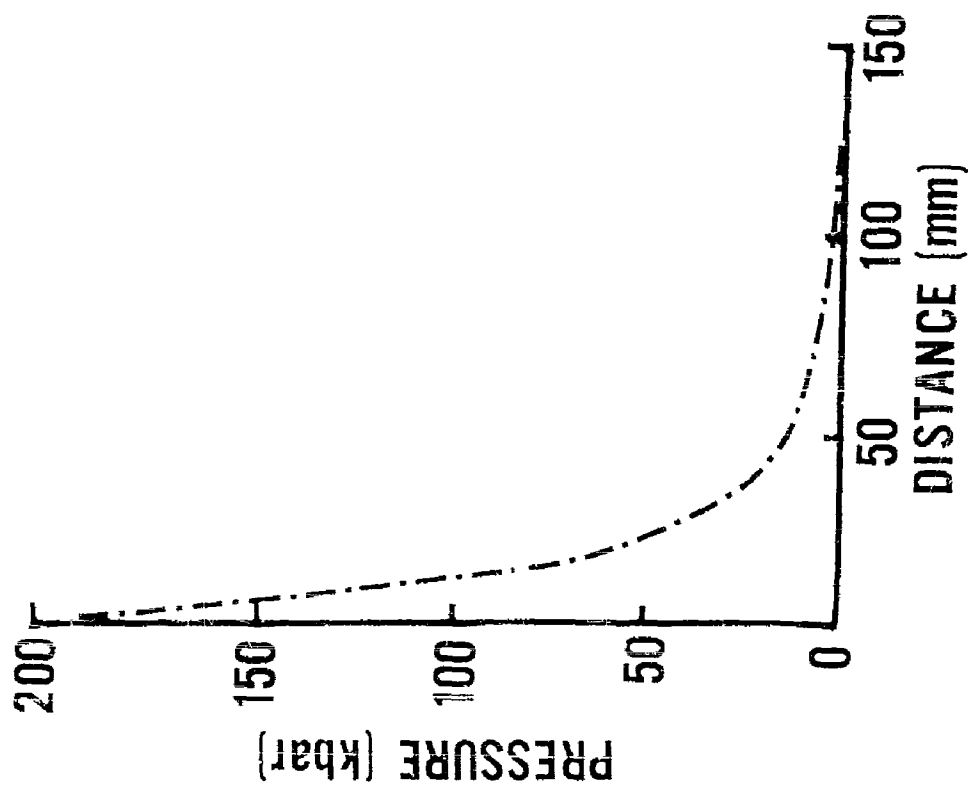


Figure 12

# SHOCK PRESSURE IN POLYURETHANE AS A FUNCTION OF THE SHOCK VELOCITY

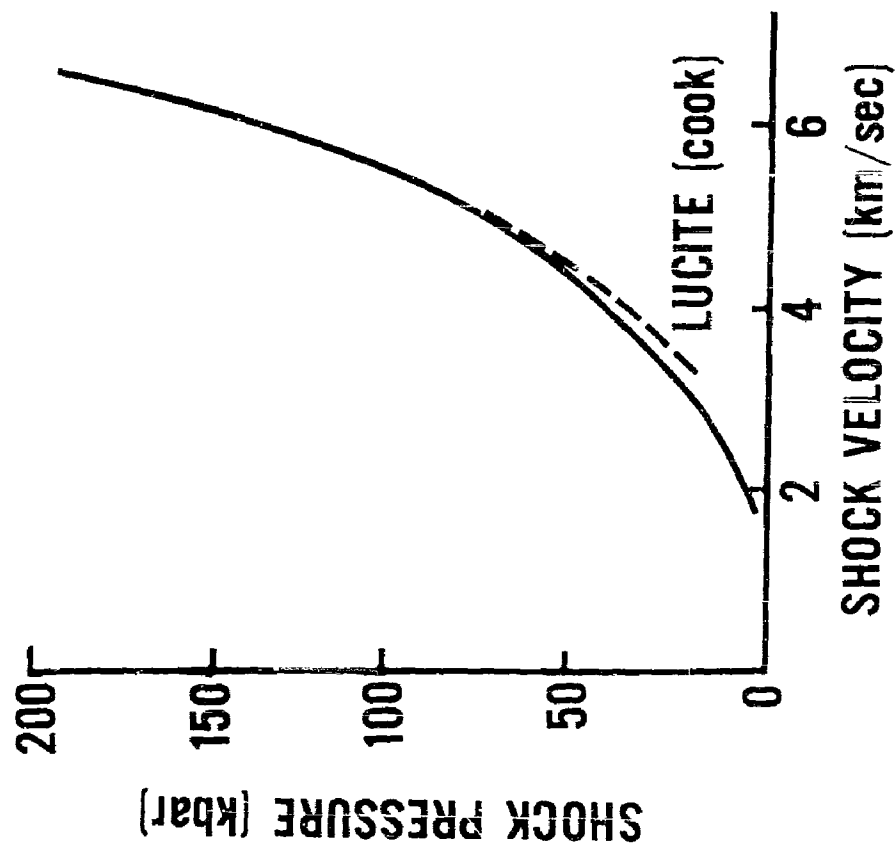


Figure 13



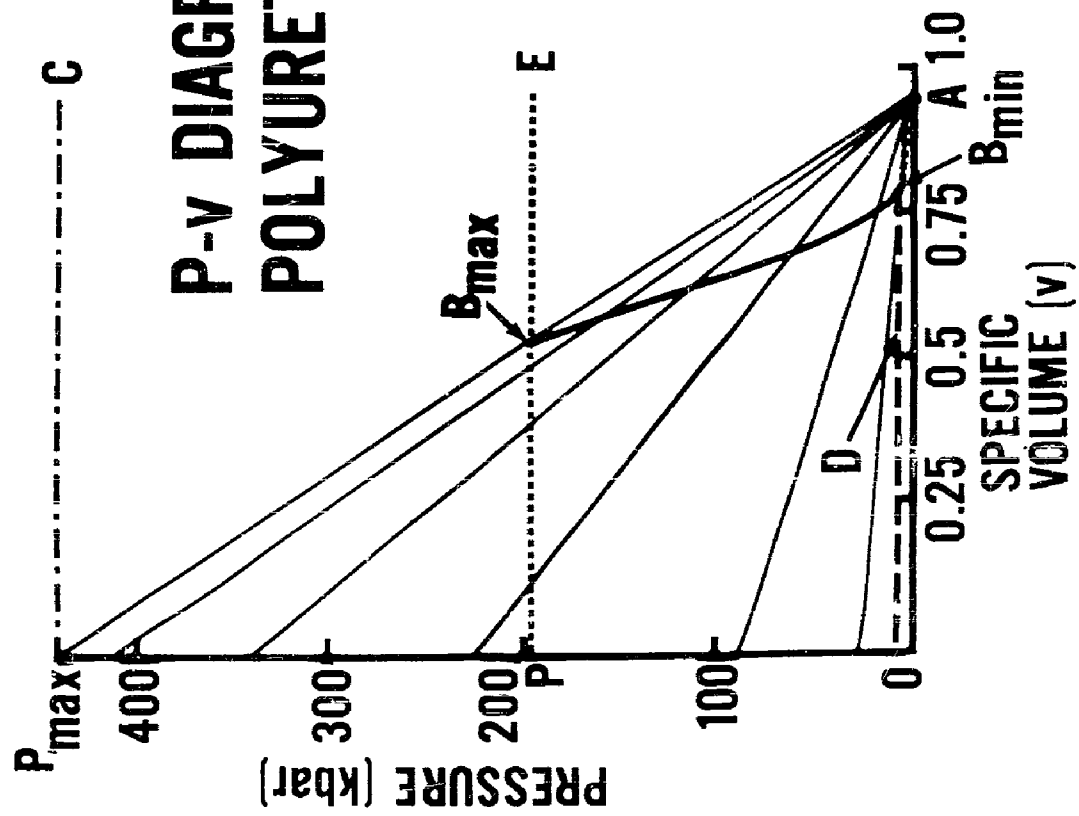


Figure 14

### Effect of Container Material and Size on Nitroglycerin Load Capacity:

Table I lists the various container materials which were tried. Containers with a 10-cm diameter were fabricated with 12.5-mm inside diameter holes and were exploded with 5 g of nitroglycerin. The aluminum and the medium-density polyethylene were the only containers to withstand the explosions. The aluminum container also withstood the explosion of 10 g of nitroglycerin with very little deformation, whether in inner or outer diameter.

The polyethylene container also withstood the explosion of 10 g of nitroglycerin with very little outside deformation (Figure 15). However, there was some deformation in both the inner and outer diameter with a 15 g load of nitroglycerin. The container sustained the detonation very well, and no fracture lines could be seen. It is evident that the expansion of the container hole, during the explosion, increases in depth and acts for a longer period of time on the bottom of the container than on the top of the container, as there the explosion gases can escape faster. The circles on Containers 1 and 2 show the original sizes of the holes before testing. For Container 3 the original size is drawn on the paper beside the tested container. To better see the bulging of the hole, this container was cut in two pieces.

Larger polyethylene containers, 30 cm in diameter, were tested with 100 and 250 g of nitroglycerin with very little outside deformation (some inner deformation). With a 500-g nitroglycerin load, the container was deformed to some extent (Figure 16) but sustained the detonation well. The circles on top of each container indicate the original sizes of the holes.

Polyethylene is a good material for explosive safety containers. It has the mechanical properties which suit the containment of an explosion. It has a high percentage of elongation (up to 600%) before break and a relatively high tensile strength. It has very good cold-flow properties and deforms rather than fractures even at high strain rates.

Polyethylene has an arrangement of threadlike molecular carbon chains which is intermediate between those of the two conventional types; one the completely ordered or crystalline arrangement and the other a purely random or amorphous one. With these two arrangements, the polymer is both tough (from its crystalline qualities) and deformable (from its amorphous arrangement). Ordinary polyethylene has a high degree of

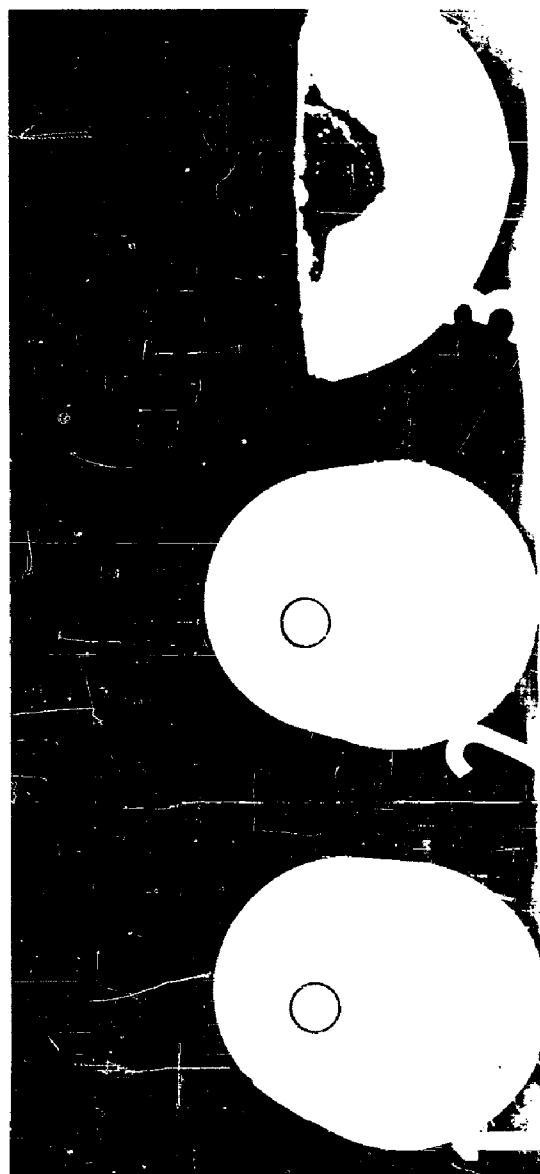
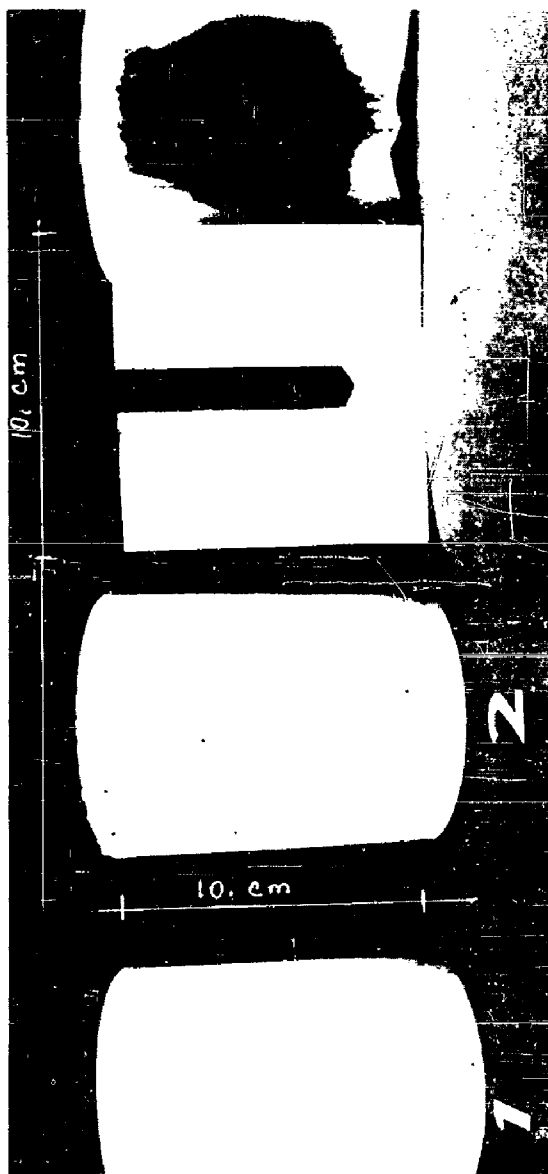


Figure 15 - Polyethylene containers tested with No. 1, 5 g nitroglycerin;  
No. 2, 10 g nitroglycerin; and No. 3, 15 g nitroglycerin.

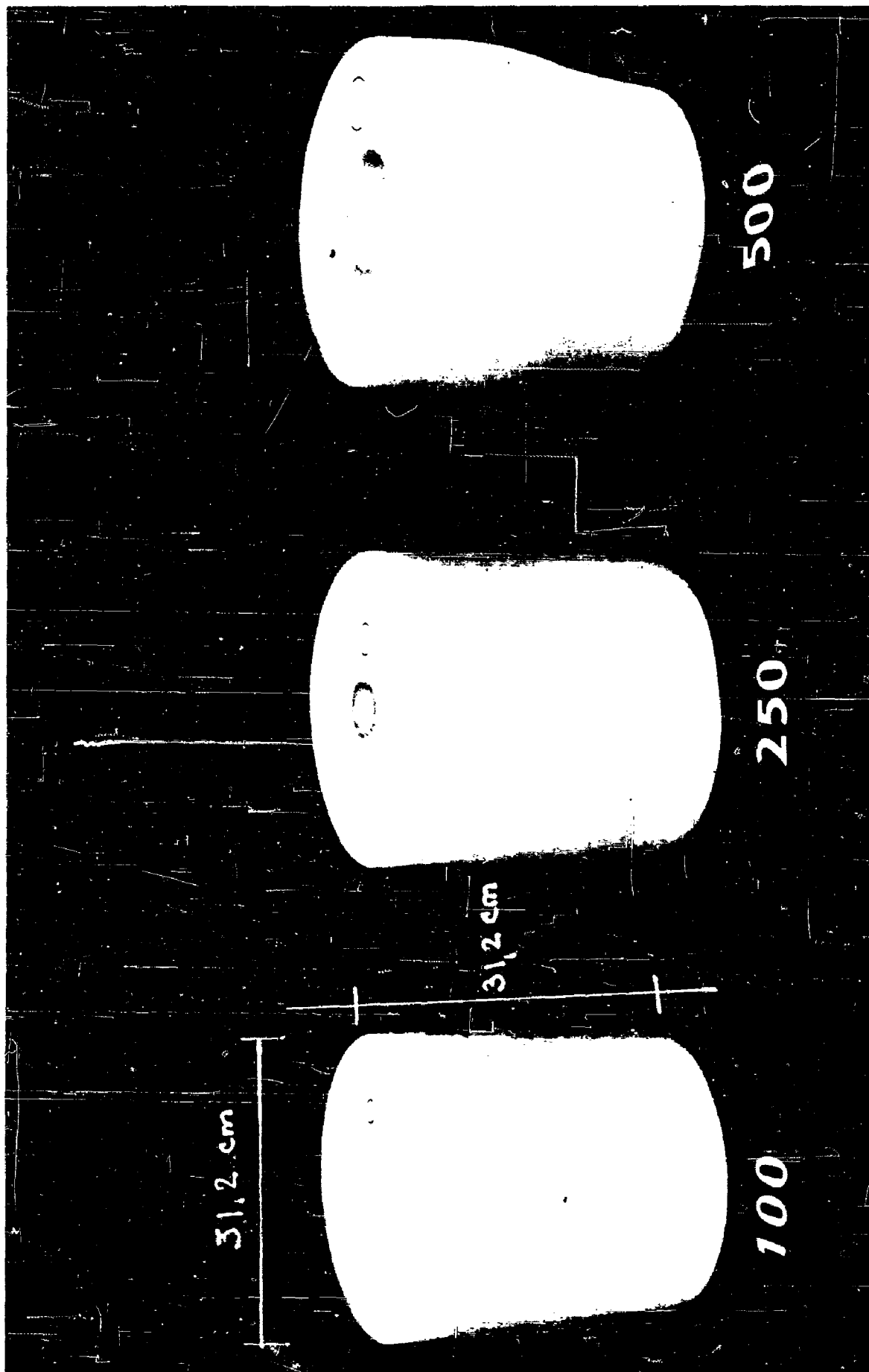


Figure 16 - Polyethylene safety container tested with 100, 250, and 500 g nitroglycerin

crystallinity (around 60%-70%) as it has been estimated from X-ray studies.

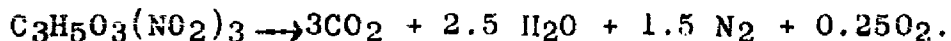
Explosions of high loads on polyethylene cause inter-molecular slippage and result in permanent deformation. This slippage causes an improvement in the alignment of the molecular chains, particularly of the continuous molecular chain, and causes an increase in the proportion of crystallinity. This alignment plus this increase in crystallinity causes the material to exhibit enhanced strength. Therefore, after the polyethylene has become extended to some degree, the material becomes stronger. With a dropping inner pressure in the container vial, caused by the escape of hot gases from the open top and the expansion of the gases, the container can withstand the explosion of relatively large amounts of explosive material.

During most of this study, the nitroglycerin was initiated to a "low-order explosion" by a Du Pont S-67 squib jolted to detonation by passing 12.5 joules of electricity through it. This was done as any accidental explosion would be activated by a relatively small quantity of energy in the form of shock, electro-static discharge, local high temperature, etc.

It is of interest, however, to determine how the barricade withstood a high-order detonation. Figure 17 shows the 10.1 cm-diameter polyethylene explosive safety container after detonating 5 and 10 g of nitroglycerin to high order by a Hercules J-2 blasting cap (12.5 joules). The J-2 blasting cap has a 1-g pentolite load. The container withstood, essentially, the detonation of a 6-g load (5-g nitroglycerin and 1-g explosive in blasting cap) with some deformation. The 10-g nitroglycerin-loaded container (+J-2 blasting cap) ruptured along the weaker side.

#### Evaluation of the Safety Containers:

Estimation of the Pressure Inside the Containers: Assuming that 5 g (3.123 cm<sup>3</sup>) of nitroglycerin are brought to explosion, the decomposition is as follows:



Applying the ideal gas laws, 5 g of nitroglycerin delivers 3.6 liters of non-dissociated gases at 0°C and 1 atm. The average molecular weight ( $\bar{M}$ ) of the combustion products is 31.32 g/mole. Assuming that the gases retain the same volume as the liquid at the moment of explosion, that the explosion temperature (T) is 4918°K, and that there is no dissociation of the reaction gases, the ideal gas pressure (P) is  $2.06 \times 10^4$  atm. However,



Figure 17 - Polyethylene containers after testing with nitroglycerin  
detonating at high order

since there is a partial dissociation of the molecules at this temperature, the pressure actually is higher. Taylor reports an explosion pressure of  $9.79 \times 10^4$  atm.

Lateral Expansion of the Container Caused by an Explosion:  
To investigate the lateral expansion of the container walls, polyurethane containers with the following dimensions were used: 8.1-cm diameter and 8.1-cm height, and 1.25-cm diameter and 4.0-cm height for the inserted vial. The expansion of the containers was recorded by means of a high-speed camera. The increase of the diameter was measured from the photographs for various nitroglycerin loads. In Figure 18 the changes in radii of several containers during the explosion of various nitroglycerin loads are given. As expected, the bulging of the container increases with increasing nitroglycerin load. These curves represent the changing of the containers radii. The acceleration of the outer wall of the container from zero to the average expansion velocity occurs within 175 usec. The resulting figures for the acceleration are given in Table II.

Lateral Pressure on the Container Walls: The pressure peaks on the container wall were measured with special piezo gages connected with an oscilloscope. Figure 19 is a sketch of the two setups used for these measurements. In setup B there is no possibility for expansion of the container and, therefore, the recorded pressure peaks are much higher, by approximately a factor of 10, than in setup A. As expected, the pressures on the bottom of the container were higher than those of the side walls. Some of the pressures are listed in Table II. In Figure 20 the pressures are plotted as a function of the nitroglycerin load. The difference,  $170 \text{ kg/cm}^2$ , between the downward pressure and the sideward pressure is caused by the thrust which is specifically caused by the ejected gases. Two measurements on polyethylene containers are also given in Figure 21 and show that the sideward pressure is lower than on polyurethane containers.

During this investigation an interesting fact was observed. It was found that the resistivity of a container (observed on polyurethane) is also influenced by the position of the container. The first polyurethane containers were found to withstand safely the explosion of about 3 g of nitroglycerin, while a container loaded with 5 g of nitroglycerin was fractured in several pieces. Usually, the containers were tested with the vial in an upright position. When the container was laid on its side, the damage to the container was much less. Five grams of nitroglycerin were exploded without fracturing the container, and it moved only a few meters.

# EXPANSION OF THE CONTAINER DURING EXPLOSION OF VARIOUS NITROGLYCERIN LOADS

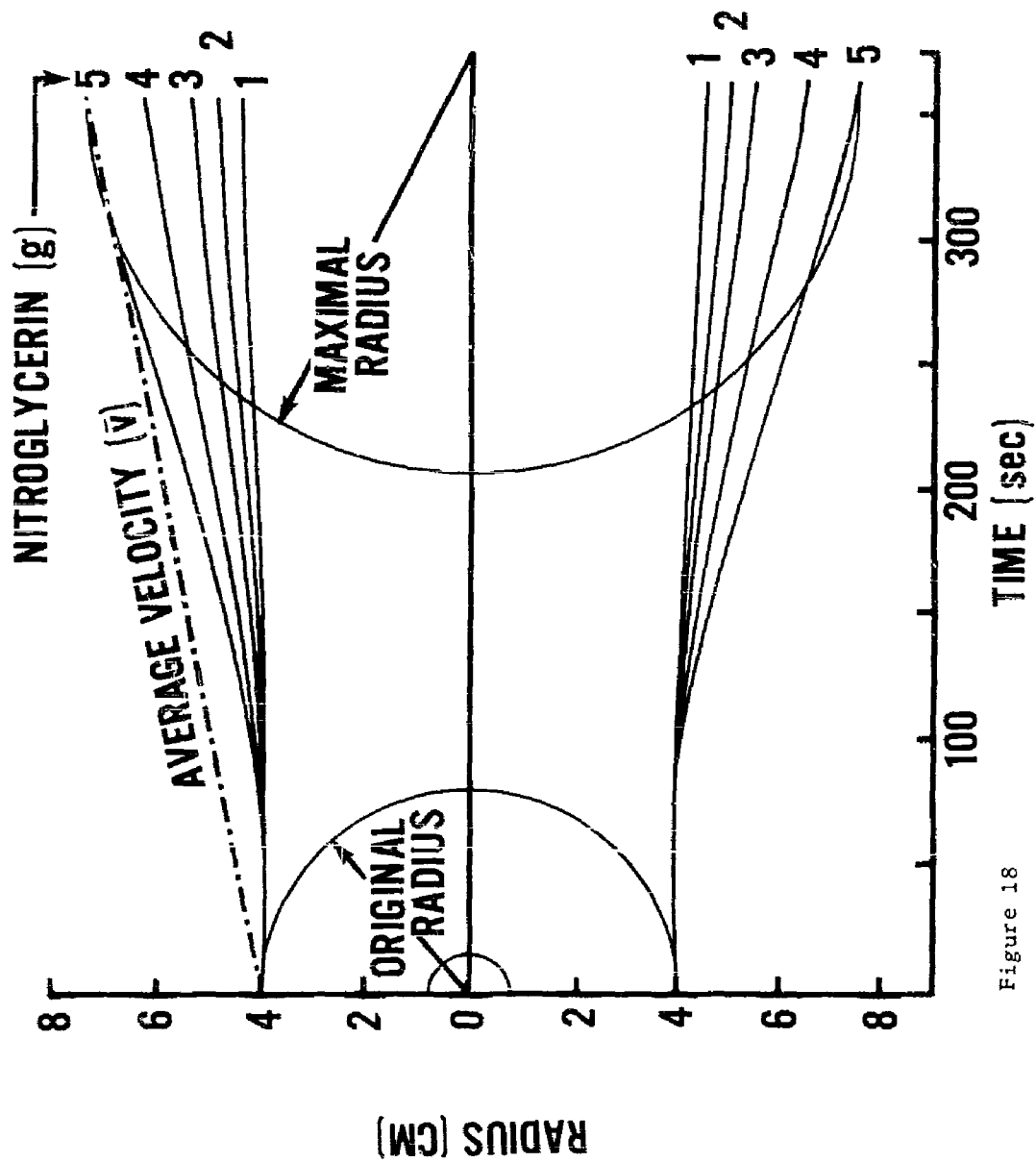


Figure 18



# **SETUP FOR PRESSURE MEASUREMENT ON THE OUTSIDE WALL OF THE CONTAINER**

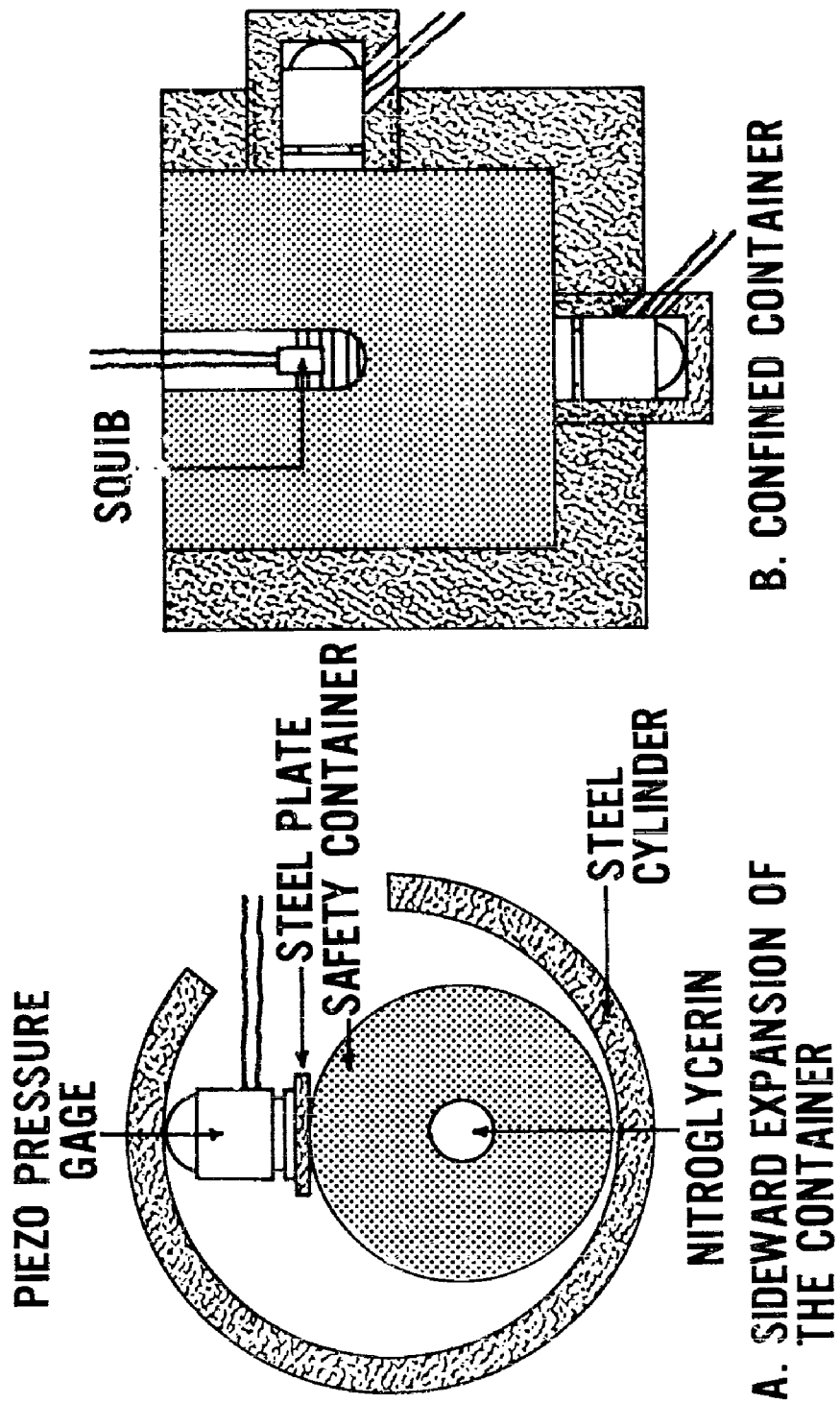


Figure 19

# PRESSURE MEASUREMENTS ON SAFETY CONTAINERS

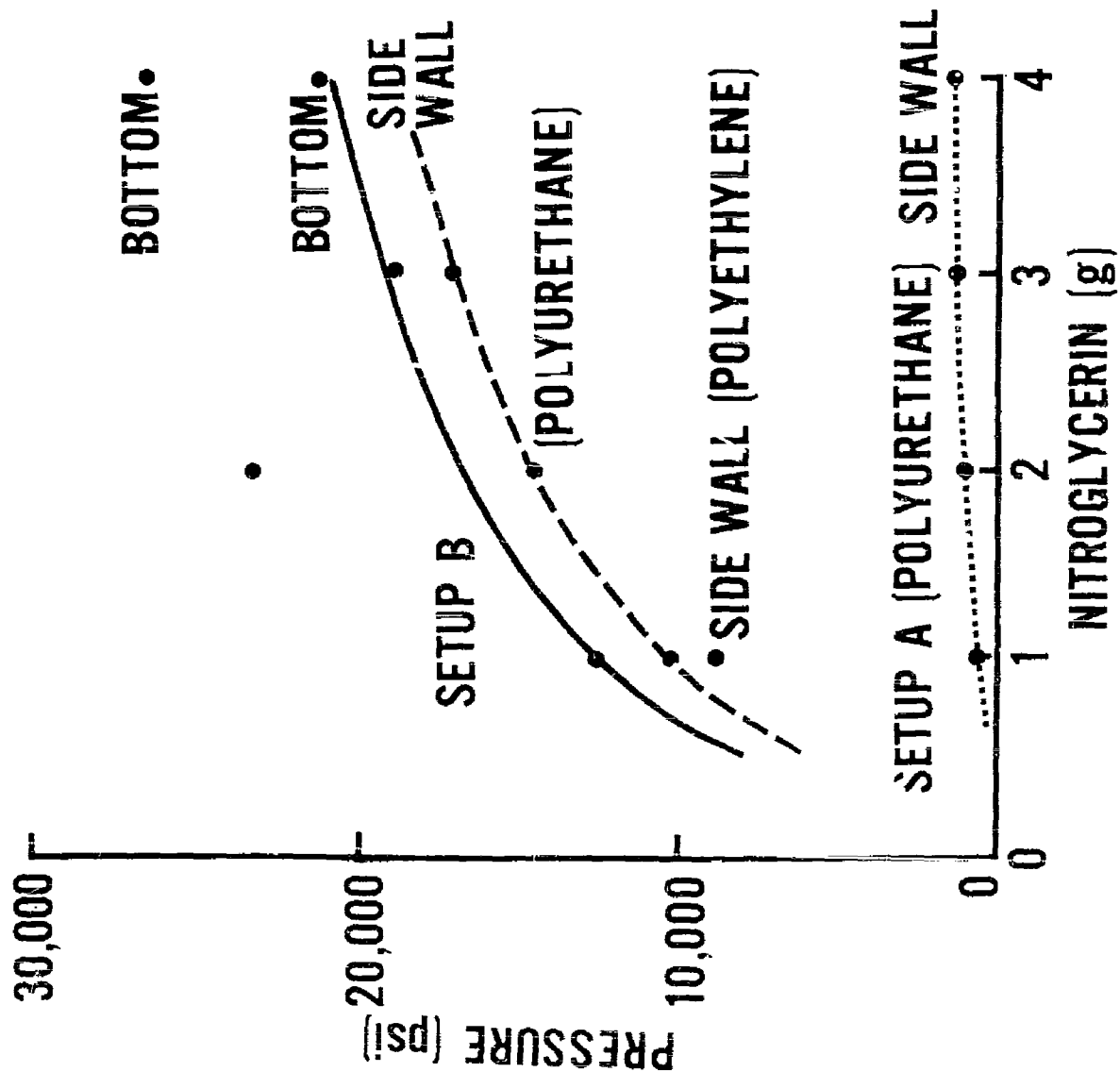
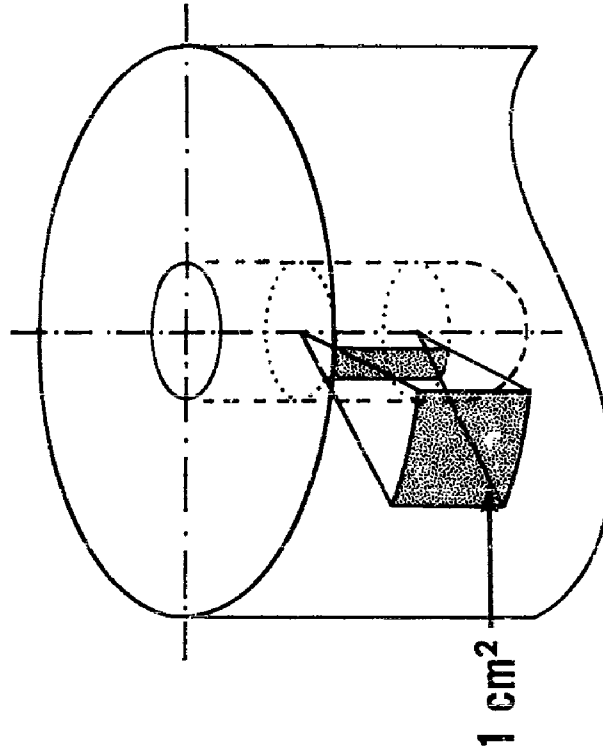


Figure 20

# CALCULATION OF THE LATERAL PRESSURE



$$P = \frac{F}{A} = \frac{ma}{A} = \frac{w}{g} \cdot \frac{a}{A}$$

THE PRESSURES CALCULATED BY THIS METHOD ARE

1 g nitroglycerin	$P = 20.1 \text{ Kg/cm}^2$
2 g nitroglycerin	$P = 32.8 \text{ Kg/cm}^2$
3 g nitroglycerin	$P = 46.8 \text{ Kg/cm}^2$
4 g nitroglycerin	$P = 86 \text{ Kg/cm}^2$
	$P = 120 \text{ Kg/cm}^2$

Figure 21

Some pressure measurements were made using strain gages in conjunction with a Wheatstone bridge and oscilloscope. The pressures obtained by this method are listed in Table III.

The lateral pressure on a container was also calculated from the mass acceleration, which has been determined from photographs of the bulging of the container. It is assumed that the mass behind each square centimeter of the outer wall surface for the height of the inserted tube is equal to the weight per gram of the section as shown in Figure 21. The weight of this section of the container is 2.086 g, inner diameter is 1.25 cm, outer diameter is 8.1 cm, and density of polyurethane is 1.043 g/cc. The decrease of web thickness, causing a decrease of weight,  $w$ , of material behind each square centimeter, and the increase of the surface,  $A$ , during the expansion of the container walls, are not considered.

With the insertion of the acceleration values for each load of nitroglycerin tested, the pressures on the outer walls of the containers can be estimated using the equation

$$P = \frac{F}{A} = \frac{ma}{A} = \frac{w}{g} \times \frac{a}{A}$$

$$m = w/g.$$

Where

- $m$  = mass of wall material
- $a$  = acceleration of wall mass
- $P$  = maximum pressure
- $w$  = weight of material per square centimeter of wall surface
- $g$  = gravity acceleration

The calculated pressures are lower than those measured with setup A in Figure 20. In this setup the container is sustained on two diametrically opposed points so that the container is restricted slightly to its free expansion. In setup B, where no expansion of the container is possible, the highest pressure values are obtained.

### CONCLUSIONS

According to the investigation on safety containers the following conclusions can be drawn.

- (1) The blast of an explosion in the container is directed upward. A load of 2 g of nitroglycerin produced a flame about 30 cm in diameter and height on the top of the container.

(2) The explosion is followed by a bulging of the container walls, which occurs with the acceleration of some thousand g's.

(3) The lateral pressures are dependent on the load of explosive and on the expansion possibilities of the container.

(4) The downward pressure is greater than the lateral one because of the downward thrust. The difference between the two figures is about  $\Delta P = 170 \text{ Kg/cm}^2$  at the explosion of 5 g of nitroglycerin.

(5) During the explosion, mechanical vibrations of the container material were recorded in the range of 50 Kc/sec.

(6) The container in an upright position did not withstand as much of an explosive charge as a container lying on its side.

(7) In polyethylene containers, less lateral pressure was measured than in polyurethane containers. Of all the materials tested, polyethylene is recommended to be used as safety container material.

(8) To achieve optimum safety for the container, the following procedures in handling the containers are recommended.

(a) The load of the container should always relate to the size of the container. The maximum load for a container of 8 cm in diameter and an inside vial of 1.25 cm is approximately 2 to 3 g for polyurethane and 5 to 10 g for polyethylene.

(b) Since the hot gases are almost completely ejected from the container, there is no protection in the area above the container. If the container, for any reason, is carried by hand, it should be kept at eye level.

(c) It is recommended that the safety containers not be carried by hand. They should be carried in special fabricated boxes with appropriate long handles.

(d) Some considerations should also be given to the downward thrust.

(e) Working with safety containers does not preclude the wearing of safety glasses.

TABLE I  
LIST OF TESTED CONTAINER MATERIAL

	Container diameter (cm)	Hole diameter (cm)	Length of Hole (cm)	Load of NG <sup>1</sup> (g)
Polyurethane	8.1	1.25	4.0	(+) 3
Polyurethane strengthened with Dacron line	8.1	1.25	4.5	(+) 5
Polyurethane encased with steel	8.1	1.25	4.0	(+) 5
Polyurethane encased with aluminum	7.5	1.25	5.0	(+) 10
Polyethylene	10.0	1.25	6.5	(+) 5
(medium density)	10.0	1.25	7.0	(+) 10
	10.0	1.35	7.0	(+) 15
7075-T-6 aluminum	8.7	1.25	6.0	(+) 10
Nylon	10.1	1.25	5.0	(-) 5
Lucite	10.1	1.25	5.0	(-) 5
Polystyrene (crosslinked)	10.1	1.25	5.0	(-) 5
Polyvinyl chlorite	10.1	1.25	5.0	(-) 5
Polypropylene	10.1	1.25	5.0	(-) 5
Polyethylene	31.2	2.3	17.0	(+) 100
	31.2	3.06	22.1	(+) 250
	31.2	5.08	16.5	(+) 500
Teflon	10.1	1.25	6.0	(-) 10

1 (+), did withstand the explosion of the indicated amount of nitroglycerin; (-) did not withstand the explosion of the indicated amount of nitroglycerin

TABLE II

EXPANSION OF SAFETY CONTAINERS DURING EXPLOSION  
OF VARIOUS AMOUNTS OF NITROGLYCERIN

Expansion Parameters	Nitroglycerin (g)				
	1	2	3	4	5
Container radius before explosion (cm)	4.0	4.0	4.0	4.0	4.0
Radius at maximum expansion (cm)	4.6	5.0	5.4	6.5	7.6
$\Delta R$ (cm)	0.6	1.0	1.4	2.5	3.6
$\bar{v}$ , average expansion velocity (m/sec)	17.1	28.6	40.0	71.5	103.0
$\bar{a}$ , average acceleration (m/sec <sup>2</sup> ) $\times 10^4$	9.8	16.3	22.9	40.9	59.4
Acceleration ( $\times 10^3 g$ )	9.9	16.6	23.4	41.7	60.4

TABLE III

MAXIMUM LATERAL AND DOWNWARD PRESSURE ON POLYURETHANE  
SAFETY CONTAINERS MEASURED AND CALCULATED BY DIFFERENT  
METHODS

Method	Load Nitroglycerin (g)					Pressure kg/cm <sup>2</sup>
	1	2	3	4	5	
Calculation eqn. (2)	20.1	32.8	46.8	86	120	lateral
Strain gauges	24		48	68	---	lateral
Setup A	35.2	73.1	85.4	94.9	---	lateral
Setup B	721	1015	1135	---	---	lateral
Setup B	975	----	1287	1483	---	downward

FILM  
on  
NIKE SPRINT SITING TEST

F. E. Hart  
U. S. Army Missile Command  
Huntsville, Ala.

Introduction

The prospect of deployment of the NIKE X Anti-Missile System in defense of the United States makes necessary the determination of the ground radius effects of an accidental explosion of a missile within a launch cell. This data is necessary because it is anticipated that the NIKE X System will be located within, or near, heavily populated metropolitan cities. Real estate costs could be prohibitive to the program unless proper design of the launch complex provides adequate safety to adjacent areas.

Tests have been planned to provide safety and design information on fragment dispersion and the ground radius effects of an explosion. This data will permit the establishment of proper quantity-distances to assure that acceptable damage risk criteria is assigned to the NIKE X sites.

A test plan was prepared by the NIKE X Project Office and submitted to the Army Materiel Command and the Armed Services Explosives Safety Board for approval.

The first test, using a half linear scale charge utilizing TNT as the explosive medium, was conducted on 18 January 1964. This test was conducted to establish a calibration reference for subsequent tests which will use NIKE SPRINT propellant instead of high explosives. A second test with SPRINT propellant will be conducted in the near future and will also be a half linear scale model under identical conditions as the first test; using TNT.

Film

Test information and data are contained in the technical report, subject: "Ground Radius Effect of Cylindrical Charge Detonated Within a Test Cell," dated 12 March 1964. A copy of this report may be obtained through Army Materiel Command channels or is available in most technical libraries.



## HAZARDS OF SLURRIES AT HANDLING VELOCITIES

By

C. B. Dale

Safety Department  
U. S. Naval Propellant Plant  
Indian Head, Maryland

In the manufacture of double-base propellant, slurry casting has been employed for a number of years, and at least two or three manufacturers are using this process at the present time. At the Naval Propellant Plant, double-base slurry casting is practiced as a part of the Inert-Diluent Process. The slurry consists of a mixture of solid ingredients (such as oxidizers and fuel) and the plasticizer (such as nitroglycerin). In the composite propellant field it is the usual method to cast motors with a slurry propellant.

Examples of composite motors that have been slurry cast at the Naval Propellant Plant are the SUBROC, the Tartar, and the Arcite Terrier. A typical arrangement for conventional slurry casting involves transferring the mix from a vertical mixer bowl through a vertical transfer line to a casting mold. The flow is controlled by a diaphragm valve and deaeration takes place through a slit plate. A vacuum is usually maintained in the mold. During the vacuum transfer of the slurry into the mold, under a high-pressure differential, an explosion hazard could exist.

Another arrangement consists of transferring the slurry from a mixing bowl to a transfer vessel under a pressure differential into a vacuum vessel. An explosion could occur due to the slurry impacting on a metal plate.

Two propellant formulations have been examined in the work to be described. They are a high-energy composite-modified double-base propellant developed by the Allegany Ballistics Laboratory known as DYN, and a composite formulation developed by the Atlantic Research Corporation known as Arcite for use in the Terrier Sustainer. The sensitivities of these materials are as follows:

	Impact (mm)	Friction (psi)	Electrostatic discharge (joules)
DYN slurry	100	4-15	12.5
Arcite	150	700	-
TNT	600+	-	-

## SLURRY GUN TESTS

The equipment used consists of a 5-cubic-foot reservoir of compressed nitrogen feeding to a solenoid valve. The solenoid valve admits pressure up to 135 psi to the gun barrel in which the slurry sample has been placed. Two diameters of gun barrels were used—a 0.83-inch (ID) and a 1.86-inch (ID). For each diameter, two barrel lengths were available—a 2-foot and a 10-foot size. A flow diagram of the equipment is shown in Figure 1. The diagram also shows the electronic timer and photoelectric cells which were used to determine the slurry velocities. Figure 2 shows the timer and photoelectric cells in position for taking measurements, and Figure 3 shows the gun in position directed at the impact plate. The gun was calibrated for velocity with dummy slurry wrapped in Saran and dummy slurry placed in cylindrical polyethylene containers.

The calibration curves shown in Figures 4 and 5 indicate that maximum velocities of 600 ft/sec and higher could be obtained.

The primary slurry tested was the DYN formulation. One size sample was 22.5 grams and the velocity was varied from 143 ft/sec to 435 ft/sec. As shown in Table I, 20 samples were tested and only 2 samples did not ignite. There may have been some ignition in these two samples but not enough to ignite the entire quantity of propellant.

The slurry of DYN, which was wrapped in Saran, came out of the barrel initially as a fine spray and subsequently one or several slugs of propellants left the barrel. The result was an ignition. This is shown in Figures 6, 7, and 8, which are photographs taken with a high-speed camera at an estimated film speed of 4000 frames/second. The velocity of the propellant was 400 ft/sec.

An attempt was made to concentrate the flow of slurry by forcing it out of a hole one-fourth inch in diameter in the lid of a polyethylene cylinder. The cylinder, in turn, was placed in the barrel.

Figures 9, 10, and 11 show the improvement possible with the new method using a dummy propellant. The estimated film speed was 4000 frames/second. The velocity of the slurry was estimated from the photographs and found to be about 200 ft/sec. With a slurry of DYN the new method gives a higher percentage of ignitions for the same initial sample weight and velocity.

A comparison of the ignitions obtained with initial sample weights of 11.5 grams of DYN slurry and 22.5 grams, as shown in Table II, shows a larger percentage of ignitions for the larger samples. The explanation for

these results is believed to be the increased probability of the presence of large clusters of slurry, i.e., one-eighth inch or one-fourth inch in diameter. This is further borne out by the results for the 115-gram samples.

Another variable investigated was the effect of vacuum. The equipment used is shown in Figure 12. It was found that at 10 mm Hg, in the vacuum receiving vessel, the 11.5-gram samples were completely consumed as shown in Table III. At a higher pressure of 60 mm Hg, there was no reaction.

For the 115-gram samples, there was one sample with complete consumption of the DYN slurry as shown in Table IV, and destruction of the receiving vessel with the other sample. It can be concluded that in vacuum the high-energy composite-modified double-base propellant is more sensitive than at atmospheric pressure. The reason may be the sensitivity of the nitroglycerin vapor.

The results of the tests with the Arcite slurry are shown in Table V. All tests were negative even when starting with 115-gram samples at a velocity of 600 ft/sec.

If the factors which are believed to be of importance in effecting the ease of ignition of slurry propellants were tabulated, they would be as follows:

- (1) The weight (and size) of the propellant projectile
- (2) The velocity of impact
- (3) The presence of a vacuum
- (4) The viscosity of the slurry
- (5) The temperature.

## DISCUSSION OF RESULTS

The lowest velocity at which ignition was obtained was 140 ft/sec using an initial slurry weight of 22.5 grams. It can be calculated that the kinetic energy of 3.5 grams of DYN slurry moving at 140 ft/sec is equal to the energy input from the 5-kilogram weight used in the drop or impact test for DYN propellant. In slurry casting, a typical velocity is in the order of 0.2 ft/sec and it would take a large quantity of slurry performing as a projectile at that velocity to have the energy required for ignition. Normally, such a situation cannot exist. To cite another case, though hopefully a rare case, 4 pounds of slurry moving at 5 ft/sec would have the necessary energy to be ignited.

It might be visualized that such a condition could exist in case the diaphragm valve controlling the transfer of slurry was opened too wide or if a plug in the casting line was released suddenly and the slurry struck an elbow in the line.

It is, therefore, recommended that the type of test described here be performed in all cases where slurry casting is carried out.

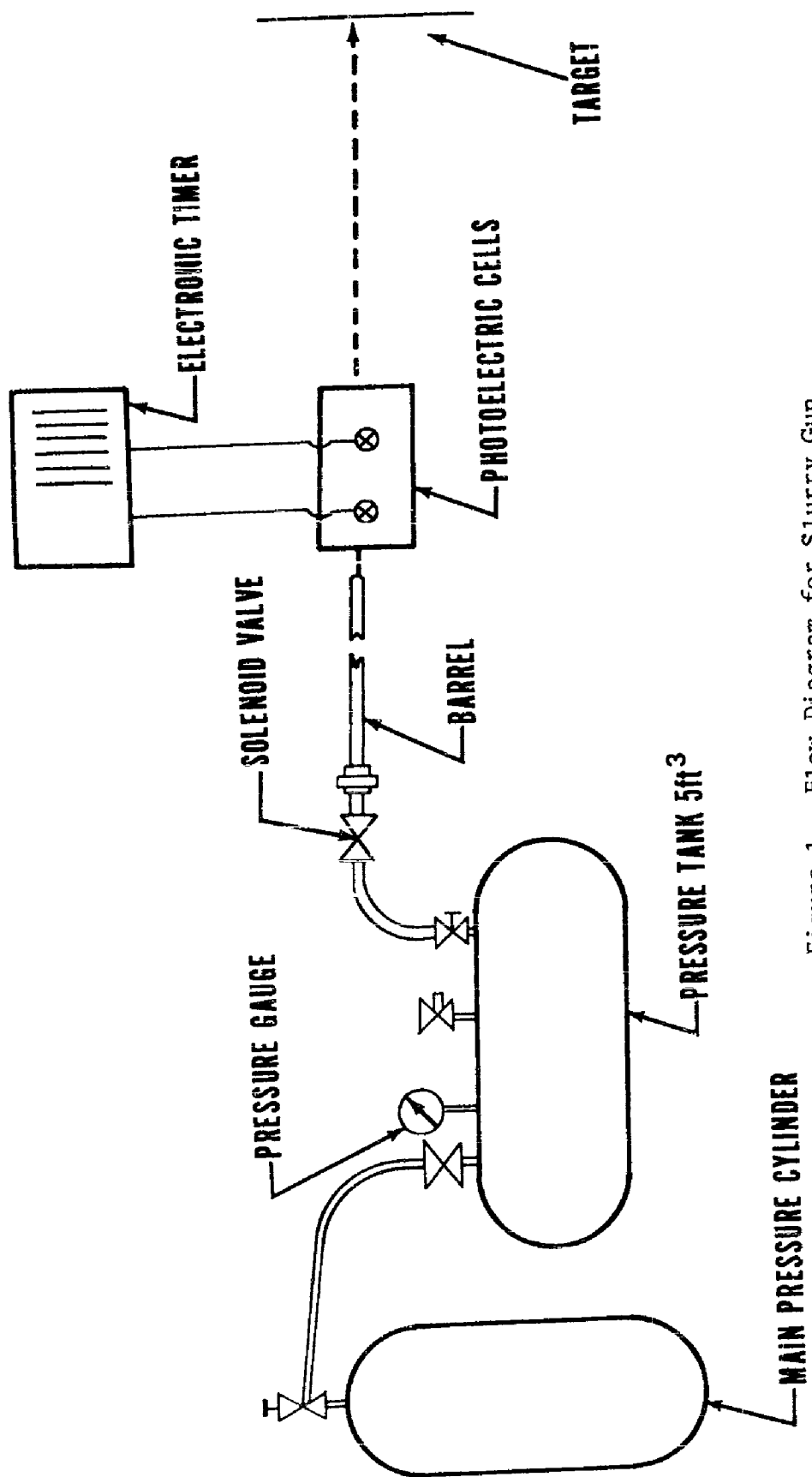


Figure 1. Flow Diagram for Slurry Gun

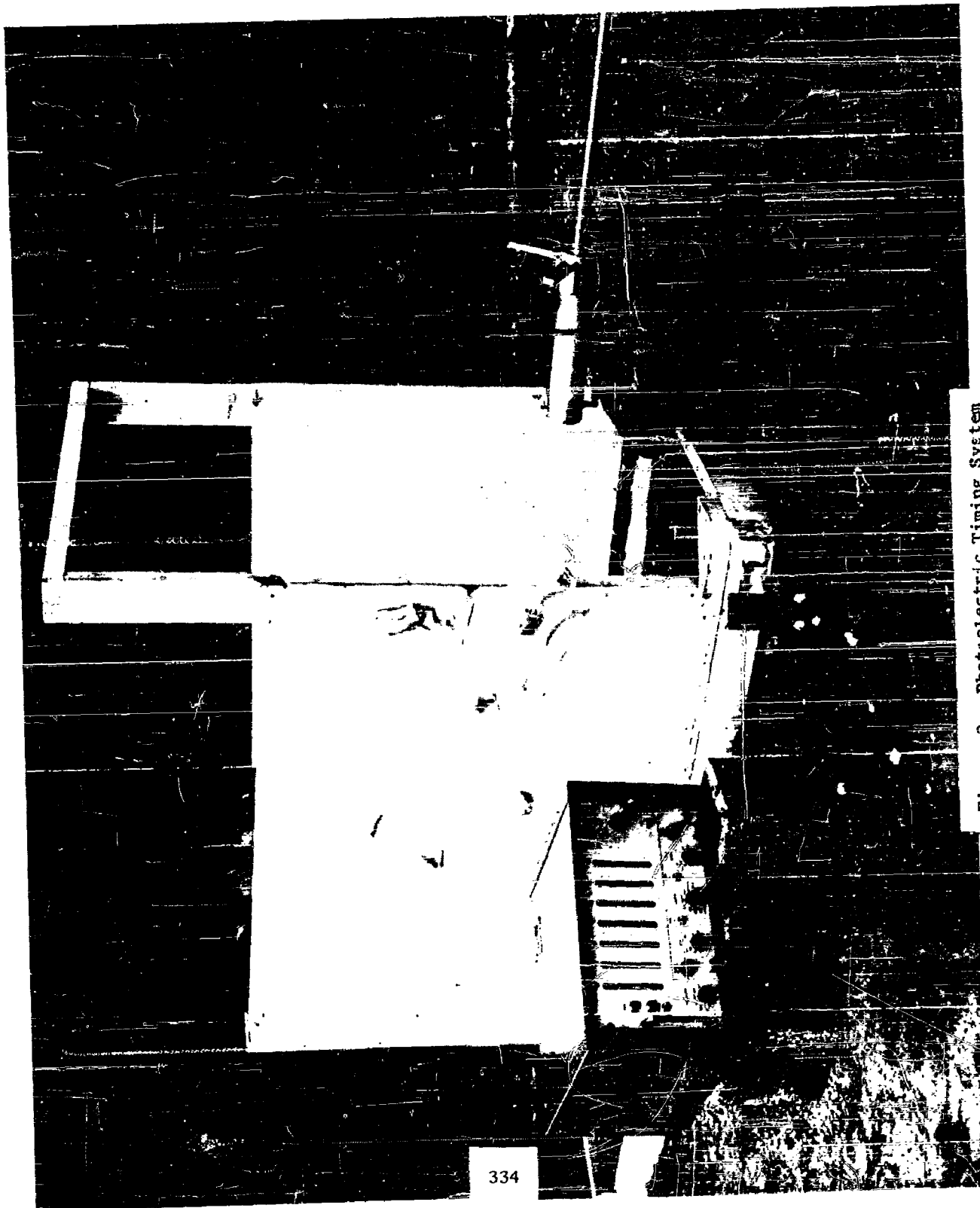


Figure 2. Photoelectric Timing System

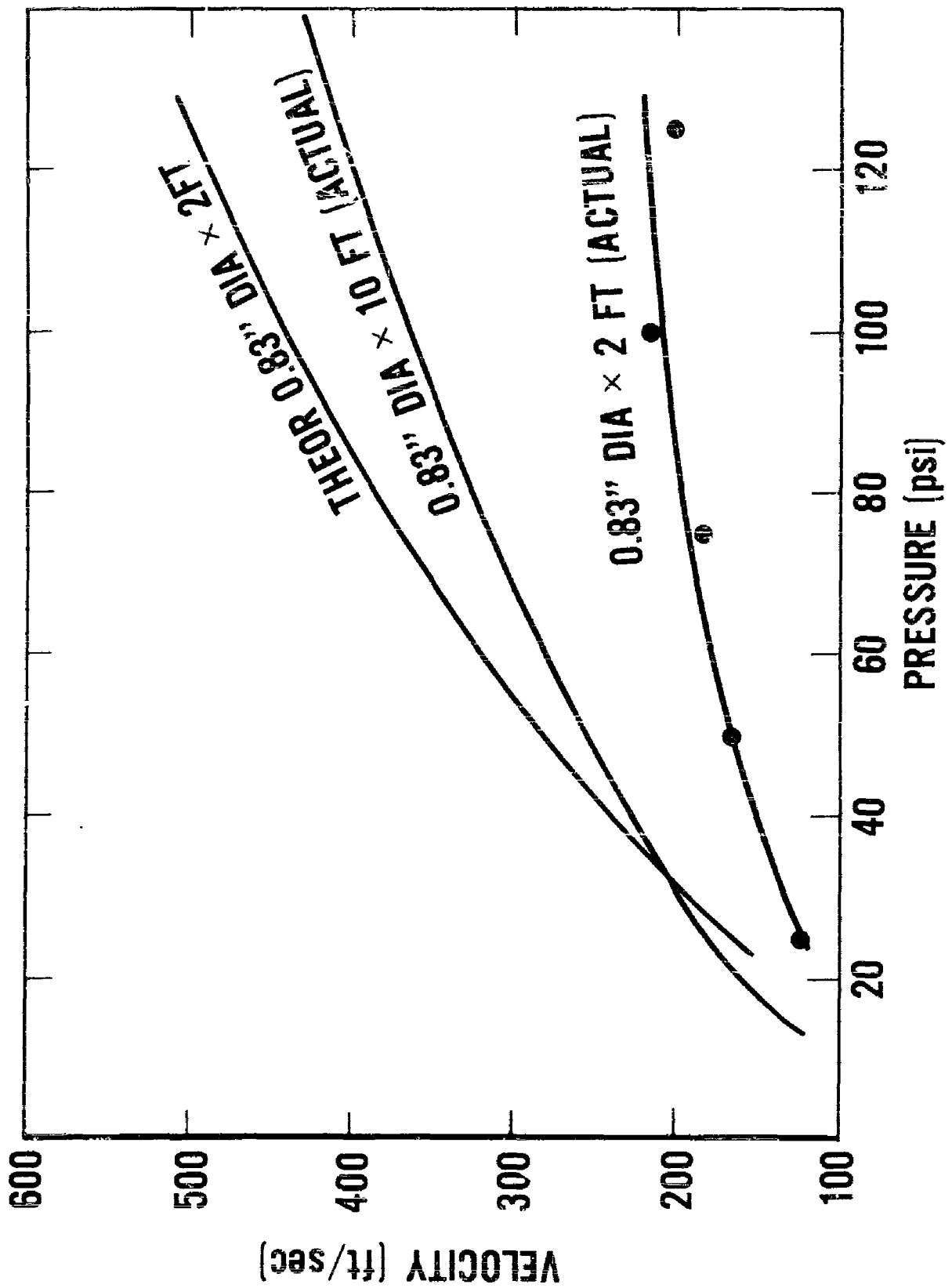


Figure 4. Gun Calibration With 22.5 Grams of Dyn Slurry

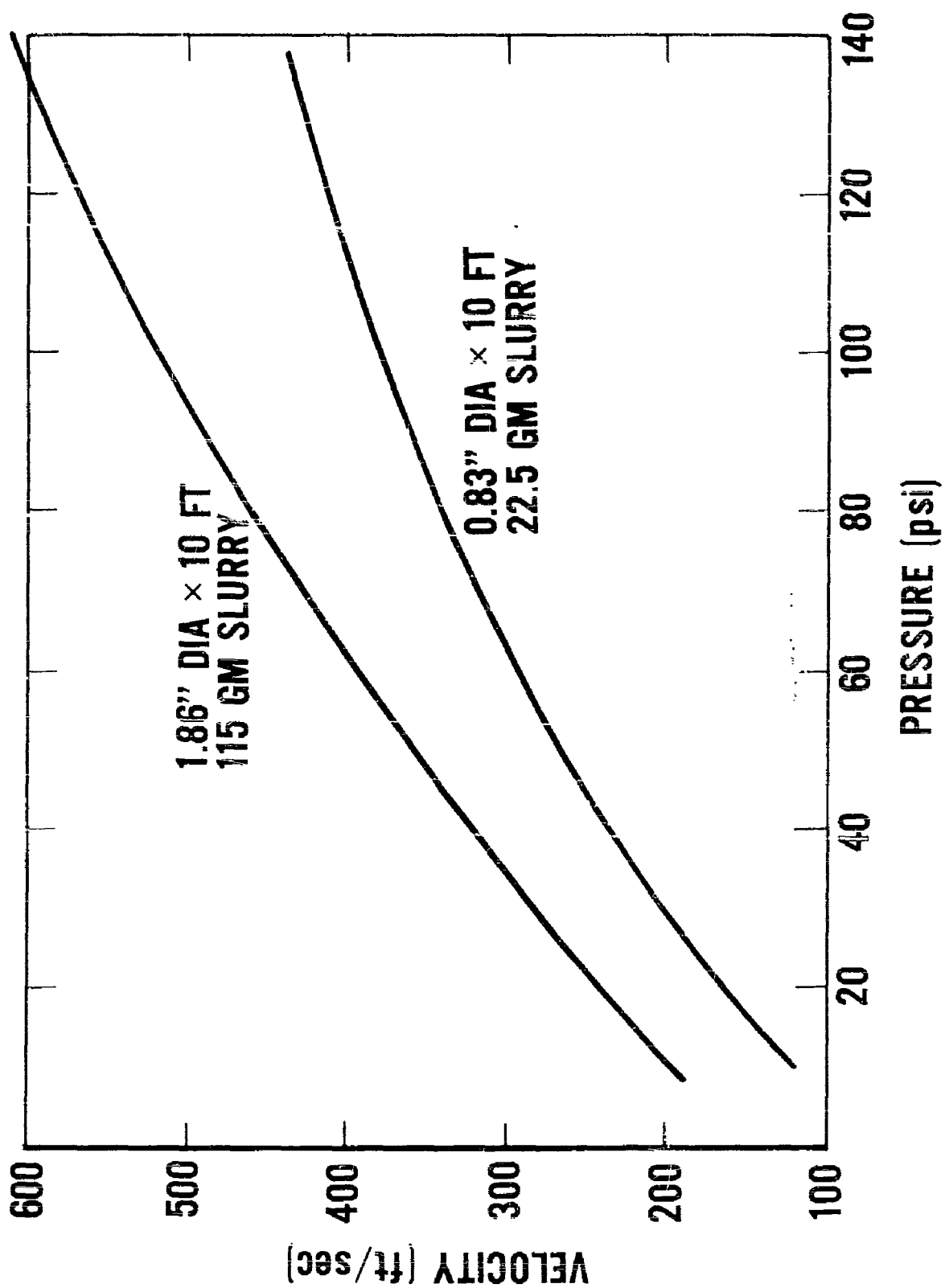


Figure 5. Gun Calibration With 115 Grams of Dyn Slurry





Figure 6. Build-up of Propellant on Impact Plate



Figure 7. Propellant Slug Approaching Impact Plate

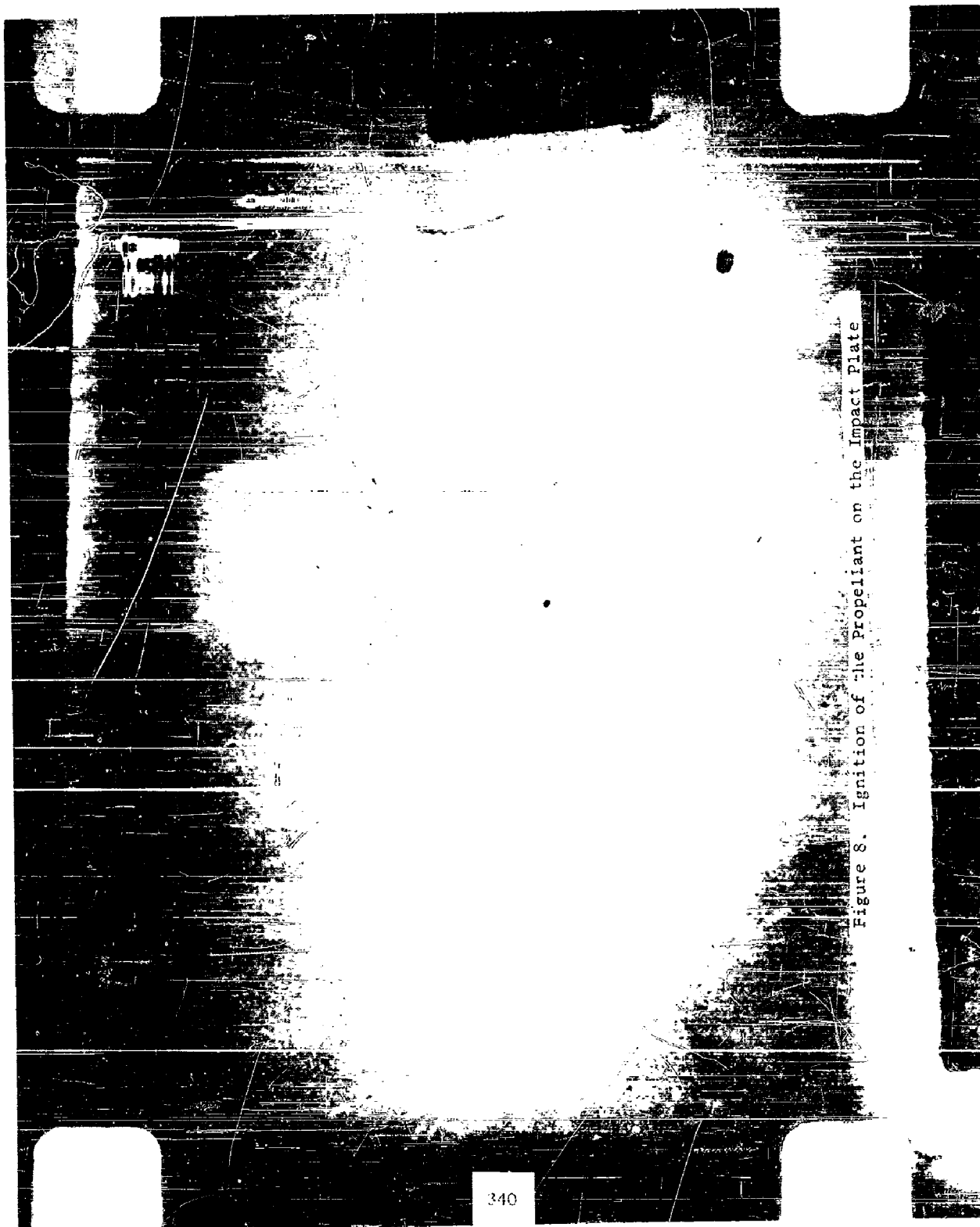


Figure 8. Ignition of the Propellant on the Impact Plate



Figure 9. Dummy Slug Leaving Muzzle



Figure 10. Dummy Slug a Short Time Later

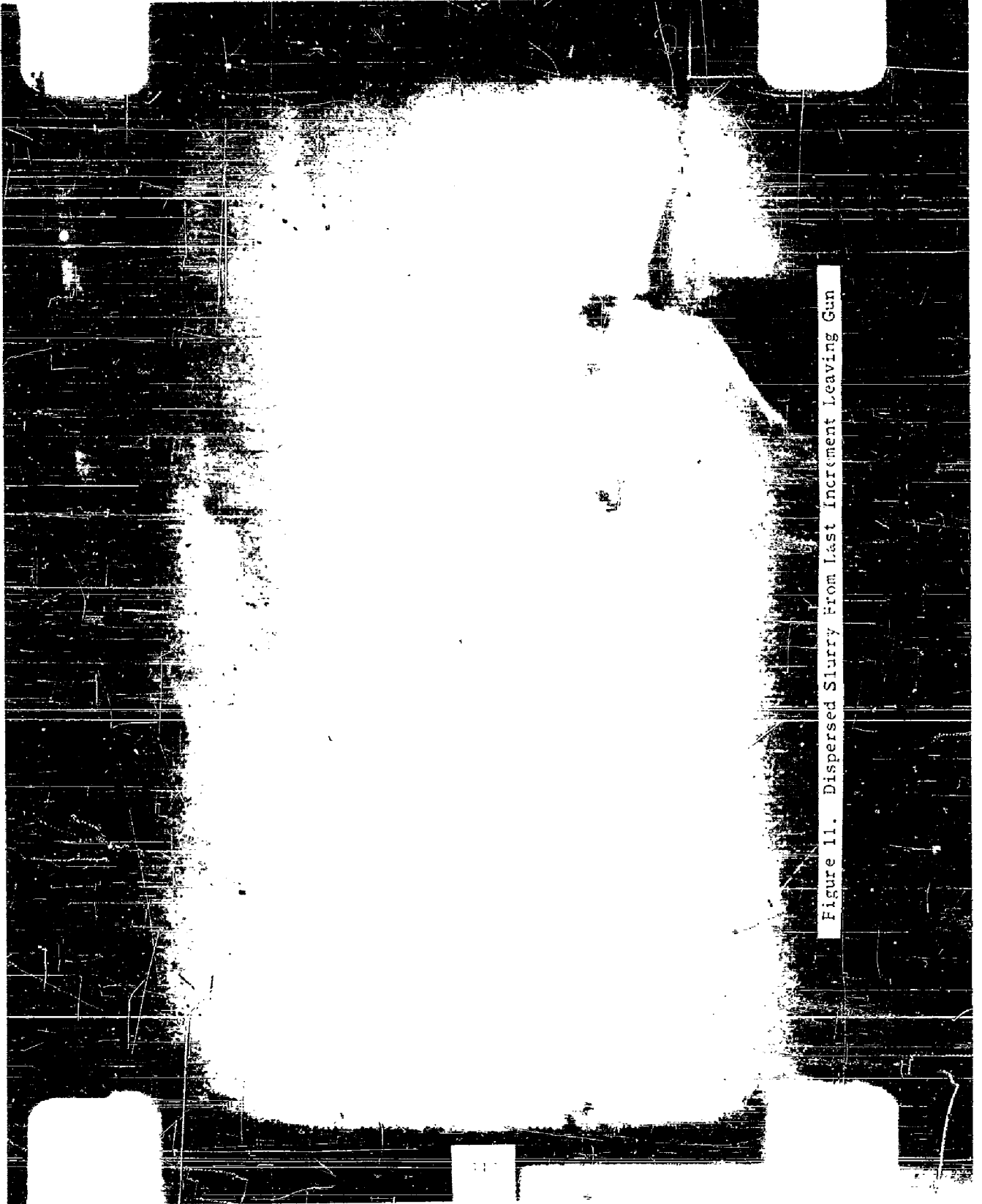


Figure 11. Dispersed Slurry From Last Increment Leaving Gun

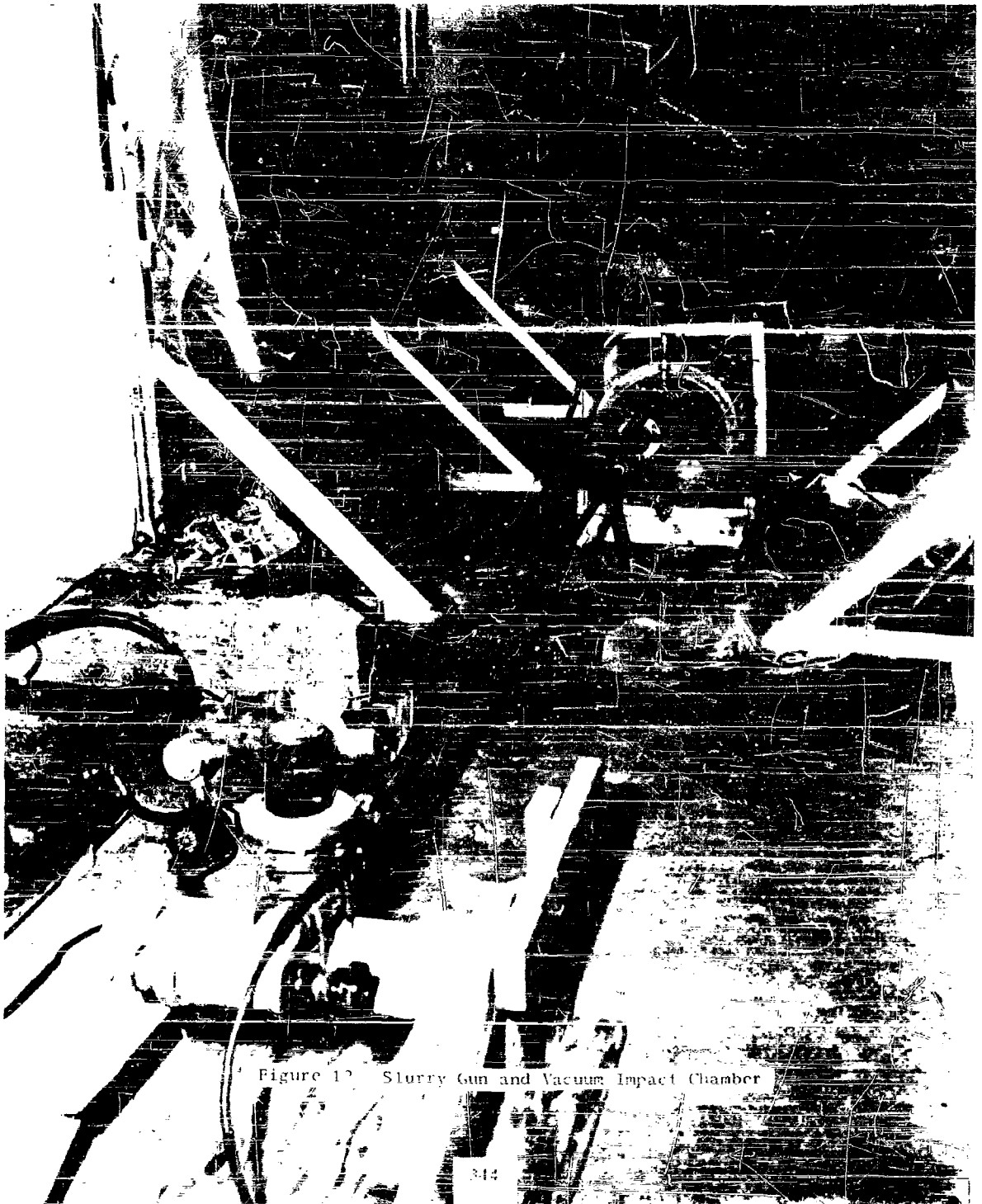


Figure 12 Slurry Gun and Vacuum Impact Chamber

Table I. Ignition Tests of 22.5 Grams Dyn Slurry (atmospheric pressure)

INITIAL PRESSURE PSI	VELOCITY FT./SEC	RESULTS			
		TOTAL RUNS	DETONATION	EXPLOSION	NO REACTION
135	435	13	—	11	2
65	301	4	—	4	—
30	202	2	1	1	—
15	143	1	—	1	—

BARREL SIZE: 0.83 IN. ID. × 10 FT  
PROPELLANT WRAPPED IN SARAN



Table II. Comparison of Ignition Tests on Dyn Slurry (atmospheric pressure)

WEIGHT (GRAMS)	INITIAL PRESSURE (psi)	VELOCITY (ft./sec.)	RESULTS			
			TOTAL RUNS	DETON- ATION	EXPLOSION	NO REACTION
11.5	135	615	15	1	3	11
22.5	135	435	13	--	11	2

BARREL SIZE: 0.83 IN. ID.  $\times$  10 FT  
PROPELLANT WRAPPED IN SARAN

Table III. Vacuum Ignition Tests of Dyn Slurry (11 - 22 grams)

WEIGHT (grams)	BARREL SIZE IN. ID × FT	INITIAL VACUUM CHAMBER PRESSURE mm	VELOCITY ft./sec	RESULTS			
				TOTAL RUNS	DETON- ATION	EXPLOSION	NO REACTION
11.5	0.83 × 10	10	200	2	—	2	—
11.5	0.83 × 10	60	200	5	—	—	5
22.5	0.83 × 10	60	143	2	—	—	2

PROPELLANT WRAPPED IN SARAN

Table IV. Vacuum Ignition Tests of Dyn Slurry (115 grams)

WEIGHT (grams)	BARREL SIZE IN. ID X FT.	INITIAL VACUUM CHAMBER PRESSURE (mm)	VELOCITY FT/SEC	RESULTS			
				TOTAL RUNS	DETON ATION	EXPLOSION	NO REACTION
115.0	1.86 × 10	14	200	1	—	1	—
115.0	1.86 × 10	50	200	1	1	—	—

PROPELLANT WRAPPED IN SARAN

Table V. Ignition Tests of Arcite Slurry (atmospheric pressure)

WEIGHT (grams)	BARREL SIZE IN. ID × FT	VELOCITY (ft./sec.)	RESULTS			
			TOTAL RUNS	DETON ATION	EXPLOSION <sup>(1)</sup>	NO REACTION
22.5	0.83 × 2	220	6	—	—	6
22.5	0.83 × 10	435	3	—	—	3
115	1.86 × 10	600	2	—	—	2

PROPELLING PRESSURE 135 PSI  
PROPELLANT WRAPPED IN SARAN

## PARTICIPANTS

Ackerman, H.	AF Systems Command, Andrews AFB, Md.
Albaugh, B.	WCMR, Mira Loma AFS, Calif.
Allovio, Jos. M.	Aerojet-General Corp., Sacramento, Cal.
Anderson, Wm. S.	Minn. Mining & Mfg Co., St. Paul, Minn.
Arbiter, Wm.	Atlantic Research Corp., Alexandria, Va.
Atkisson, Jas. B.	Jet Propulsion Lab, CIT, Pasadena, Cal.
Attaway, C. D.	Thiokol Chem. Corp., Longhorn AAP, Tex.
Austin, J. M.	Barksdale AFB, La.
Bachtell, N. D.	ASESB, Wash., D. C.
Balcom, R. E.	USNAD Bangor, Bremerton, Wash.
Beery, G. T.	AF Aeronautical Propulsion Lab, Wright-Patterson AFB, Ohio
Bobko, J. R.	Eastern Contr Mgmt Regn, Olmsted AFB, Pa.
Boston, CWO R. L.	US Army Arty Bd, Fort Sill, Okla.
Boyer, R. E.	USNOIS China Lake, Calif.
Breakefield, BG D. E.	Dir of Maintenance, USA S&M Command, Wash., D.C.
Breneman, B. D.	Rocketdyne, McGregor, Texas
Brinkley, H. L.	'US Army Mat Com Safety Agency, Charlestown, Ind.
Brown, Earl P.	Chanute AFB, Ill.
Brown, Dr. L. M.	Rohm & Haas Co., Redstone Arsenal, Ala.
Browne, T. P.	USA Missile Support Com, Redstone Arsenal, Ala.
Bruer, E. E.	Pacific Missile Range, Point Mugu, Cal.
Buchanan, H. G.	BRL, Aberdeen Proving Ground, Md.
Burdick, LCol J. R.	Hq USAF, AFSSSG, Wash., D. C.
Canipe, MSgt C. L.	Barksdale AFB, La.
Carlson, J. C. B.	Hercules Pwdr Co., Radford AAP, Va.
Carsey, J. N.	USNPP Indian Head, Md.
Caruthers, CWO A. E.	USNWS Yorktown, Va.
Chassee, Capt. A. J.	AFETR, Patrick AFB, Fla.
Colitti, O. A.	Picatinny Arsenal, Dover, N. J.
Collingsworth, K. L.	Norton AFB, Calif.
Connelly, J. W.	CNO, Dept of the Navy, Wash., D. C.
Cooke, C. R.	Edwards AFB, Calif.
Cooper, J. T.	Thiokol Chem Corp., Longhorn Div., Texas
Couch, Gerald	United Technology Ctr, Sunnyvale, Calif.
Crellin, John	R&D Directorate, AMC, Wash., D. C.
Cross, J. F.	Hercules, Allegeny Ball. Lab., Cumberland, Md.
Dale, Dr. C. B.	USNPP Indian Head, Md.
Damon, Dr. G. H.	Bureau of Mines, Dept of Interior, Wash., D.C.
Daugherty, J. H.	Aberdeen Proving Ground, Md.

Deputy, J. G.  
Diebold, J. P.  
Docherty, Bruce M.  
Donaldson, P. A.  
Drake, J. T.  
Dreyer, Capt. O. F.  
Driscoll, T. W.  
Duguid, Dr. R. H.  
Dunigan, T. E.

East, R. C., Jr.  
Eckhart, C. V.  
Economy, Gus S.  
Elrod, J. F.  
Elwell, Col. R. L.  
Ennis, G. S.  
Essick, M. L.  
Estes, C. B.  
Eichhorn, Earl  
Forsey, R. K.  
Fowler, W. T.  
Fravel, Wm. H.

Gaines, T. L.  
Gayle, J. B.  
George, Capt. W. E.  
Gibson, F. C.

Giesler, B. E.  
Gilbert, A. H.  
Goff, C. R.  
Gore, Maj. H. C.  
Graham, E. L.  
Graves, J. W.  
Green, J. F.

Grothe, J. R.  
Gunn, John W.

Hamilton, A. W.  
Hampe, J. W.

Haney, C. J.  
Hanna, R. J.  
Harbarger, J. F.

Atlantic Research Corp., Alexandria, Va.  
USNOTS China Lake, Calif.  
Asst General Counsel, OSA, Wash., D. C.  
USNOTS China Lake, Calif.  
Rem. Rand Div of Sperry Rand, La. AAP, La.  
CO, USNPP Indian Head, Md.  
Hercules Powder Co., Wilmington, Del.  
Edgewood Arsenal, Edgewood, Md.  
Picatinny Arsenal, Dover, N. J.

Barksdale AFB, La.  
San Francisco Procurement Dist., Oakland, Cal.  
Norton AFB, Calif.  
USNAD Crane, Ind.  
Hq USAF, AFHIS, Wash., D. C.  
USNOL White Oak, Silver Spring, Md.  
Rohm & Haas Co., Redstone Arsenal, Ala.  
USA Missile Com, Redstone Arsenal, Ala.  
SSD, El Segundo, Cal.  
ADC, Ent AFB, Colo.  
Radford Army Ammo Plant, Radford, Va.  
Hercules, Bacchus Works, Magna, Utah

Louisiana AAP, Shreveport, La.  
NASA, Huntsville, Ala.  
Barksdale AFB, La.  
Bureau of Mines, Dept of Interior,  
Pittsburgh, Pa.  
Edwards AFB, Calif.  
Louisiana AAP, Shreveport, La.  
Lone Star AAP, Texarkana, Texas  
Barksdale AFB, La.  
Army Procurement & Supply Agency, Joliet, Ill.  
Lone Star AAP, Texarkana, Texas  
BuWeps Fleet Readiness Rep. Pacific,  
San Diego, Calif.  
Interstate Commerce Commission, Wash., D.C.  
Johns Hopkins Univ., Applied Physics Lab.,  
Silver Spring, Md.

Hercules Powder Co., Wilmington, Del.  
Central Cont Mgmt Reg., Wright-Patterson  
AFB, Ohio  
Defense Atomic Support Agency, Wash., D.C.  
Boeing Co., Seattle, Wash.  
Thiokol Chemical Corp., Huntsville, Ala.

Hardy, W. G.	Orbital Space Stn., General Dynamics Astronautics, San Diego, Calif.
Hart, F. E.	USA Missile Com, Redstone Arsenal, Ala.
Harton, E. E., Jr.	NASA Hq, Wash., D. C.
Havron, H. C.	Thiokol, Longhorn Army AP, Texas
Hayden, W. G.	Thiokol, Wasatch Div., Utah
Hayes, J. F.	Thiokol, Denville, N. J.
Helms, E. D.	Holston Defense Corp., Holston AAP, Tenn.
Henson, Maj. E. O.	Barksdale AFB, La.
Herman, R. C.	ASESB, Wash., D. C.
Hester, J. F.	Brown Engr. Co., Inc., Huntsville, Ala.
Hicks, R. F.	Martin Co., Orlando, Fla.
Hicks, Col. S. R.	USA S&M Command, Wash., D. C.
Hilsman, Col. E. H.	ASESB, Wash., D. C.
Hinson, Paul C.	Mason-Hanger, Silas Mason Co., Iowa AAP, Burlington, Iowa
Hoffman, R. W.	American Potash & Chemical Corp., L.A., Cal.
Holley, H. Q.	Thiokol, Rocket Opns Ctr, Ogden, Utah
Hough, F. Z.	BuNavWeps, D/N, Wash., D. C.
Howard, H. W.	NavMissile Ctr, Point Mugu, Cal.
Jenkins, W. T.	Hercules Powder Co., Wilmington, Del.
Jersin, H. E. S.	Army Materiel Command, Wash., D. C.
Jezek, Lou	Army Materiel Command, Wash., D. C.
Johnson, F. H.	AF Flight Test Ctr, Edwards AFB, Calif.
Karick, F. S.	NASA Wallops Stn., Wallops Is., Va.
Kennedy, B. J.	DCASR (PT), Phila., Pa.
Kennedy, H. C.	NPP Indian Head, Md.
Kerns, A. J.	Picatinny Arsenal, Dover, N. J.
Kilgore, D. B.	AF Log Com, Wright-Patterson AFB, Ohio
King, Paul V.	NASA Kennedy SFC, Cocoa Beach, Fla.
Kohlheyer, R. C.	Douglas Aircraft, Santa Monica, Calif.
Kornegay, R. J.	Hq 25th Air Div., McChord AFB, Wash.
Krackenberger, H. F.	Longhorn AAP, Marshall, Texas
Kristoff, F. T.	Hercules, Allegeny Ballistics Lab., Cumberland, Md.
Kronick, 2d Lt J. M.	AF Plant #66, McGregor, Texas
Lecher, Wm. A.	USA Test & Evaluation Com, Aberdeen PG, Md.
Leigh, C. S.	Amcel Propulsion Co, Asheville, N. C.
Lento, L. L.	American Cyanamid Co., Stamford Research Labs., Conn.
Long, Leo	Thiokol, Longhorn, Texas
Loss, E. T.	Office, Chief of Transportation, D/Army, Wash., D. C.

Lusk, C. B.	Rocketdyne, McGregor, Texas
Lynch, J. C.	OOAMA, OOOSS, Hill AFB, Utah
Mann, B. C.	Andrews AFB, Md.
Marsh, Henry	50 Ramsey Rd., Wilmington, Del.
Marston, J. W.	La. Army Ammunition Plant, La.
Maxim, W.	NASA Lewis Res Ctr, Cleveland, Ohio
Meenan, H. J.	Aberdeen Proving Ground, Md.
Miller, Capt. H. J.	AF Systems Command, SSD, L. A., Cal.
Miller, J. A.	Olin Mathieson Chem Corp., Marion, Ill.
Miller, S. S.	Johns Hopkins Univ., Applied Physics Lab., Silver Spring, Md.
Minnich, B. H.	Rocketdyne, Canoga Park, Cal.
Molloy, J. J.	Rocketdyne, Canoga Park, Cal.
Mondano, R. L.	Custom Materials, Inc., Chelmsford, Mass.
Morris, B. W.	Aerojet-General Corp., Downey, Cal.
Murphy, J. L.	Thiokol, Huntsville, Ala.
Murphy, J. J.	Barksdale AFB, La.
Murakami, R. E.	Boeing Co., Seattle, Wash.
Mussey, R. A.	BuNavWeps, D/N, Wash., D. C.
Myers, Ray L.	AMC Fld Safety Agency, Charlestown, Ind.
McCaffrey, R. T.	St. Louis Procurement Dist., St. Louis, Mo.
McCants, Col. L. S.	Chairman, ASESB, Wash., D. C.
McCay, W. C.	Longhorn Army Ammo Plant, Texas
McElroy, S. H.	USNWL Dahlgren, Va.
McElvogue, R. A.	Thiokol Chemical Corp., Longhorn, Texas
McGahan, L. C.	Pacific Missile Range, Pt Mugu, Cal.
McGuffie, Maj. J. T.	Louisiana AAP, Shreveport, La.
McLeod, R. D.	Aerojet-General Corp., Sacramento, Cal.
McSmith, Duane	NASA Langley Res Ctr, Hampton, Va.
Napadensky, Mrs. H. S.	IIT Research Institute, Chicago, Ill.
Neal, R. E.	Jet Propulsion Lab., CalTech, Pasadena, Cal.
Nicholas, Glen	USA Missile Command, Redstone Arsenal, Ala.
Nolan, W. J.	DCASR, Allegeny Ballistics Lab, Cumberland, Md.
Oberholzer, Lt. R. J.	AFSC, SSD, Los Angeles, Cal.
Oeinck, R. H.	Lockheed Propulsion Co., Redlands, Cal.
Olsen, F. N.	Boeing Co., Seattle, Wash.
Orihood, G. M.	Aerojet-General Corp., Sacramento, Cal.
Otto, H. W.	Esso Res & Engr Co., Linden, N. J.
Owings, J. T.	USNPP Indian Head, Md.



Padgett, Harry  
Perkins, R. G.  
Pratt, W. A.  
Price, Dr. Donna N.  
Prior, L. C.

Ragsdale, R. S.  
Reid, Douglas  
Richardson, J. M.

Richardson, R. J.  
Riley, Wm. S.  
Rowland, A. A.  
Royall, R. Raymond  
Roylance, H. M.  
Rule, Frank  
Ruth, Carl R.

Sanford, TSgt R. W.  
Scannell, G. F.  
Scott, H. T.  
Settles, Jas. E.  
Shaw, Lou J.  
Shirock, R. J.  
Short, F. E.  
Shuey, Dr. H. M.  
Simmons, G. J.  
Sims, J. H.  
Sims, W. H.

Singer, Earl  
Slight, Geo. B.  
Snook, Claude  
Stanckiewicz, W. P.  
Stosz, Max J.  
Stout, Boyd C.  
Stuckey, Max T.  
Sullivan, MSgt P. R.  
Swain, R. L.

Talton, R. T.  
Thomas, Wm. B.  
Thune, H. P.  
Tullos, T. A.  
Tweed, P.

Urizar, M. J.

Thiokol Chem Corp., Brunswick, Ga.  
ASES, Wash., D. C.  
Thiokol, Longhorn AAP, Texas  
USNO White Oak, Silver Spring, Md.  
Jet Propulsion Lab, Caltech, Pasadena, Cal.

Aerospace Corp., El Segundo, Calif.  
USNOTS China Lake, Calif.  
Rem. and Div of Sperry Rand, Louisiana  
AAP, Shreveport, La.  
CONARC, Fort Monroe, Va.  
Thiokol, Longhorn AAP, Texas  
Day & Zimmerman, Inc., Lone Star AAP, Texas  
Holston AAP, Kingsport, Tenn.  
BuNavWeps, D/Navy, Wash., D. C.  
Holston Defense Corp., Holston AAP, Tenn.  
BRL, Aberdeen Proving Ground, Md.

AF Western Test Range, Vandenberg AFB, Cal.  
Thiokol, Bristol, Pa.  
Atlantic Research Corp., Alexandria, Va.  
Hercules Powder Co., Wilmington, Del.  
Atlanta Contract Mgt Dist, Atlanta, Ga.  
MOCUM, Warren, Mich.  
Harvey Aluminum Sales, Inc., Milan AAP, Tenn.  
Rohm & Haas Co., Redstone Arsenal, Ala.  
Milan AAP, Milan, Tenn.  
Thiokol, Longhorn AAP, Texas  
BuWeps Flt Readiness Rep Atlantic,  
USNAS Norfolk, Va.  
Picatinny Arsenal, Dover, N. J.  
USNAD Crane, Ind.  
USNWS Concord, Calif.  
Army Proc & Supply Agency, Joliet, Ill.  
USNOL White Oak, Silver Spring, Md.  
AF Missile Dev Ctr, Holloman AFB, New Mex.  
Thiokol Chemical Corp., Elkton, Md.  
AF Western Test Range, Vandenberg AFB, Cal.  
NASA Langley Res Ctr, Hampton, Va.

Atlantic Research Corp., Alexandria, Va.  
USA Missile Command, Redstone Arsenal, Ala.  
Aberdeen Proving Ground, Md.  
Thiokol Chem Corp., Marshall, Texas  
Avco Corp., Wilmington, Mass.

Los Alamos Scientific Lab., Los Alamos, N.M.

Van Dolah, R. W.

Van Patten, E. W.

Voeglein, J. F.

Vogt, C. I.

Walker, J. A.

Wall, J. D.

Wall, L. C.

Walsh, M.

Ward, ..

Weals, F. H.

Wenzel, A. B.

White, R. C.

Whitmore, LCol R. D.

Wiegand, W. W.

Wigger, G. P.

Wilkenson, T. H.

Williamson, Ted G.

Wilson, A. W.

Wilson, Paul D.

Winfl, Carl

Wood, L. H.

Yeakey, R. A.

Zimmer, Dr. M. F.

Wetzell, O.

Bureau of Mines, Dept of Interior,  
Pittsburgh, Pa.

USA Munitions Command, Dover, N. J.

Edgewood Arsenal, Md.

USNPP Indian Head, Md.

Lockheed Missiles & Space Corp.,  
Sunnyvale, Calif.

Thiokol, Longhorn AAP, Texas

Picatinny Arsenal, Dover, N. J.

USNWS Concord, Calif.

Rem Rand Div of Sperry Rand, Louisiana AAP, LA

USNOTS China Lake, Calif

GM Research Labs, GMC, Ellettsville, Calif.

Gen Dynamics Astronautics, San Diego, Calif.

Longhorn AAP, Texas

Rocketdyne, Canoga Park, Calif

Chief Engineers, D/Army, Wash., D. C.

DCS/Personnel, D/Army, Wash., D. C.

Orden Contract Mgt Dist., Hill AFB, Utah

Dow Chemical Co., Midland, Mich.

Pacific Missile Range, Pt Mugu, Calif.

Aerojet General Corp., Azusa, Calif.

Thiokol, Marshall, Texas

Thiokol, Brigham City, Utah

USNPP Indian Head, Md.

Thiokol, Longhorn Div.



DEPARTMENT OF DEFENSE EXPLOSIVES SAFETY BOARD -  
2461 EISENHOWER AVENUE  
ALEXANDRIA, VIRGINIA 22331-0600

DDESB-KMC

07 JUL 2000

MEMORANDUM FOR DDESB RECORDS

SUBJECT: Declassification of Explosives Safety Seminar Minutes

References: (a) Department of Defense 5200.1-R Information Security Program, 14 Jan 1997

(b) Executive Order 12958, 14 October 1995 Classified National Security Information

In accordance with reference (a) and (b) downgrading of information to a lower level of classification is appropriate when the information no longer requires protection at the originally level, therefore the following DoD Explosives Safety Seminar minutes are declassified:

- a. AD#335188 Minutes from Seminar held 10-11 June 1959.
- b. AD#332709 Minutes from Seminar held 12-14 July 1960.
- c. AD#332711 Minutes from Seminar held 8-10 August 1961.
- d. AD#332710 Minutes from Seminar held 7-9 August 1962.
- e. AD#346196 Minutes from Seminar held 20-22 August 1963.
- f. AD#456999 Minutes from Seminar held 18-20 August 1964.
- g. AD#368108 Minutes from Seminar held 24-26 August 1965.
- h. AD#801103 Minutes from Seminar held 9-11 August 1966.
- i. AD#824044 Minutes from Seminar held 15-17 August 1967.
- j. AD#846612 and AD#394775 Minutes from Seminar held 13-15 August 1968.
- k. AD#862868 and AD#861893 Minutes from Seminar held 9-10 September 1969.

The DoD Explosives Safety Seminar minutes listed above are considered to be public release, distribution unlimited.

DANIEL T. TOMPKINS  
Colonel, USAF  
Chairman

Attachments:

- 1. Cover pages of minutes

cc:  
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MINUTES  
of the Sixth  
EXPLOSIVES SAFETY SEMINAR  
ON  
HIGH-ENERGY SOLID PROPELLANTS

Held at the  
Barksdale Air Force Base  
Shreveport, La.

on  
18-20 August 1964

Host  
Longhorn Army Ammunition Plant  
Marshall, Texas  
Armed Services Explosives Safety Board  
Washington, D. C. 20315

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